VOL. LV, NO. 6, JUNE, 1960

THE Journal OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

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THE AMERICAN LEATHER CHEMISTS ASSOCIATION
PUBLICATION OFFICE, EASTON, PA.

Fifty-sixth Annual Meeting

June 19-22, 1960

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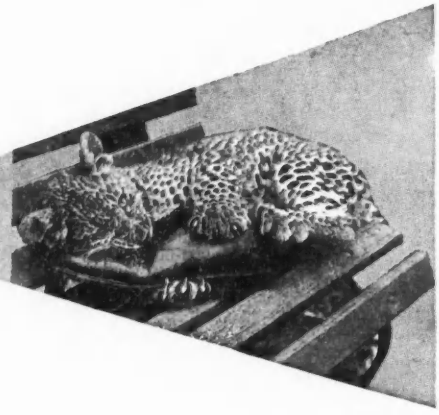

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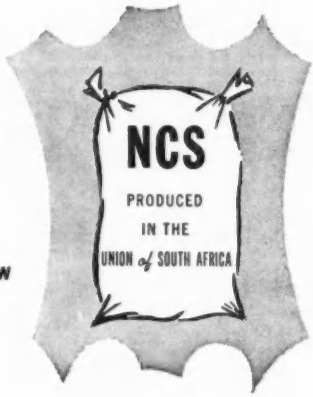

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The Journal

of the

AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. LV JUNE 1960 No. 6

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Dr. Ian Somerville, right, and Mr. Richard Fisher of Rohm & Haas are discussing the early days of zirconium tanning materials.

Dr. Somerville himself supervised tanning, in a commercial tannery, the piece of kidskin shown here. Though this was 29 years ago, the leather is still round, plump, and mellow.

Rohm & Haas first supplied zirconium tanning materials commercially as acidic solutions. Continuing development work then produced in 1938 a dry acid salt much easier to handle and use.

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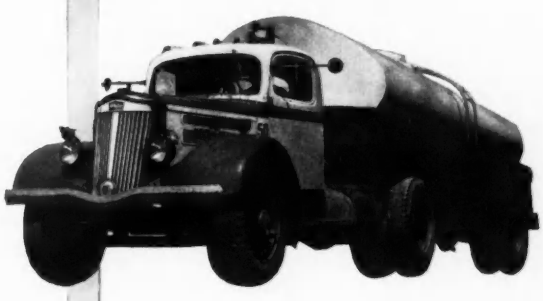

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Unloading Bulk Salt From Boxcars—at Lower Cost

While salt is a relatively low-cost material, handling it often involves considerable expense in time and manpower. Unloading bulk salt from boxcars, for example, has always been one of the problem spots. Today, however, a number of money-saving unloading methods have been developed for large and small plants. Here are four of these methods, and the equipment used in each.

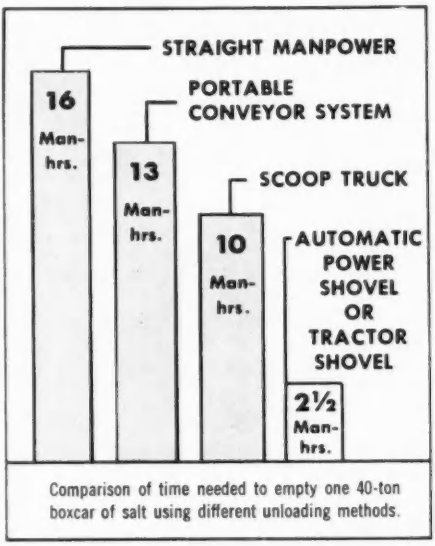
Portable Conveyor System. This is the simplest improvement over straight manpower. A power-operated belt conveyor of suitable length is placed inside the car door, and salt is then shoveled onto it. A good conveyor for most needs is generally about 16 to 18 feet long, and capable of moving salt up a 30° slope. With a conveyor, a good deal of manpower in shoveling is needed, but one 40-ton car can be unloaded in about 12 to 14 man-hours—a 19% saving in time over straight manpower.

Scoop Truck and Conveyor. A scoop truck is simply an enlarged scoop shovel with two small wheels at the bowl of the scoop. Whereas a shovel holds only about 20 lbs. of salt—a scoop truck holds 100 to 150 lbs. In operation, the scoop is pushed into the salt, tilted up, and then wheeled to the conveyor at the car door. With just one of these scoops, a worker can unload 40 tons of salt in 10 hours—and once the job is under way, there's room for two or three more men with scoops.

Automatic Power Shovel. This device consists of a large, power-operated scraper blade mounted on a cable—and is one of today's most popular devices for unloading bulk salt. The scraper is moved toward the end of the car. As soon as this motion stops, a power winch takes hold to drag the scraper toward the car door. Salt is pushed out through the door by the scraper. One man can unload a 40-ton car in 2 or 3 hours by this unique method. The installed cost of both winch and power motor is moderate.

Small Gasoline or Electric Tractor Shovel. This is somewhat similar to a fork-lift truck, but with a scoop mounted in place of the forks. One man on this type of truck can empty a 40-ton car in 2 or 3 hours—the same as power-shovel unloading. The higher cost of a tractor shovel is often justified when it can be made available for other purposes.

The best unloading method for your plant, of course, depends on the salt tonnage involved, and on the plant layout. But one of the methods just described should pay for itself—in savings on time and labor—over a relatively short period.



Comparison of time needed to empty one 40-ton boxcar of salt using different unloading methods.

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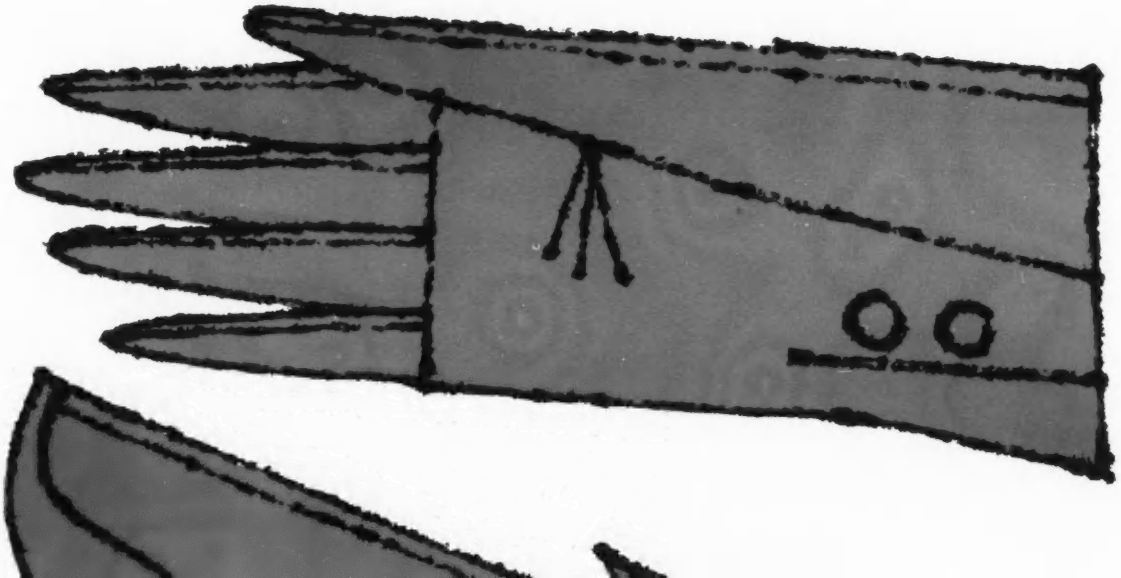

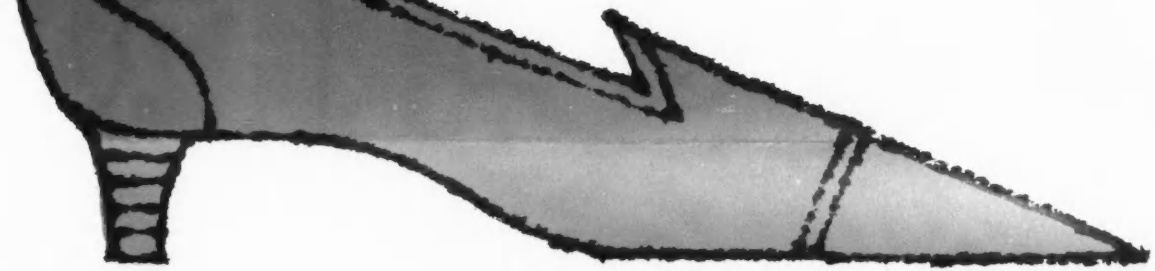

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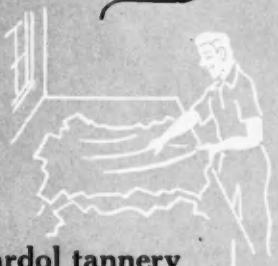

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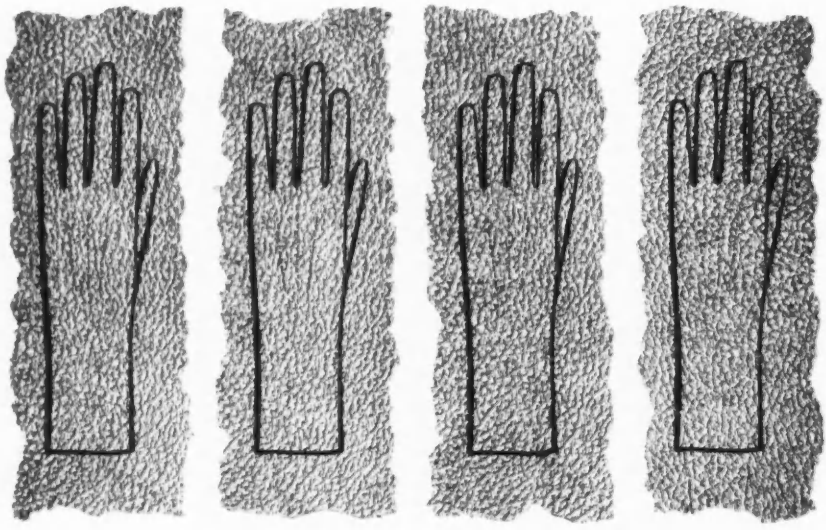

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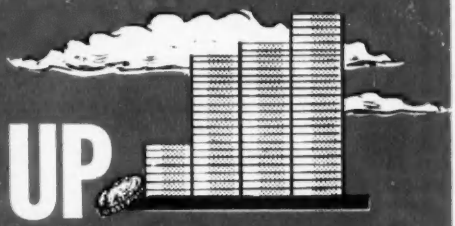

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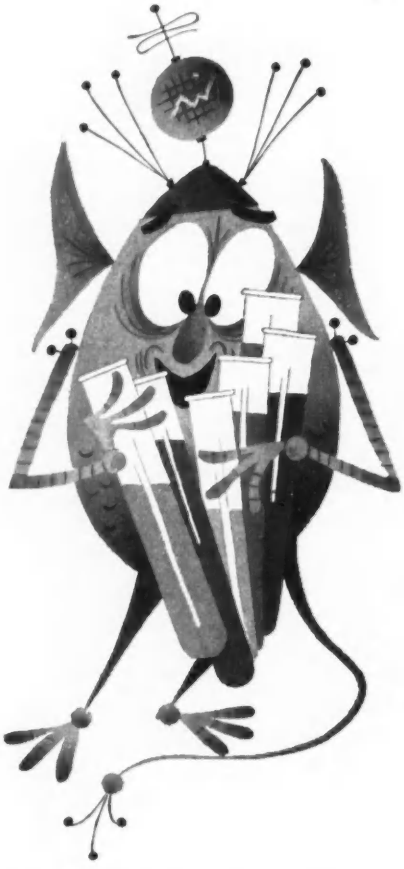

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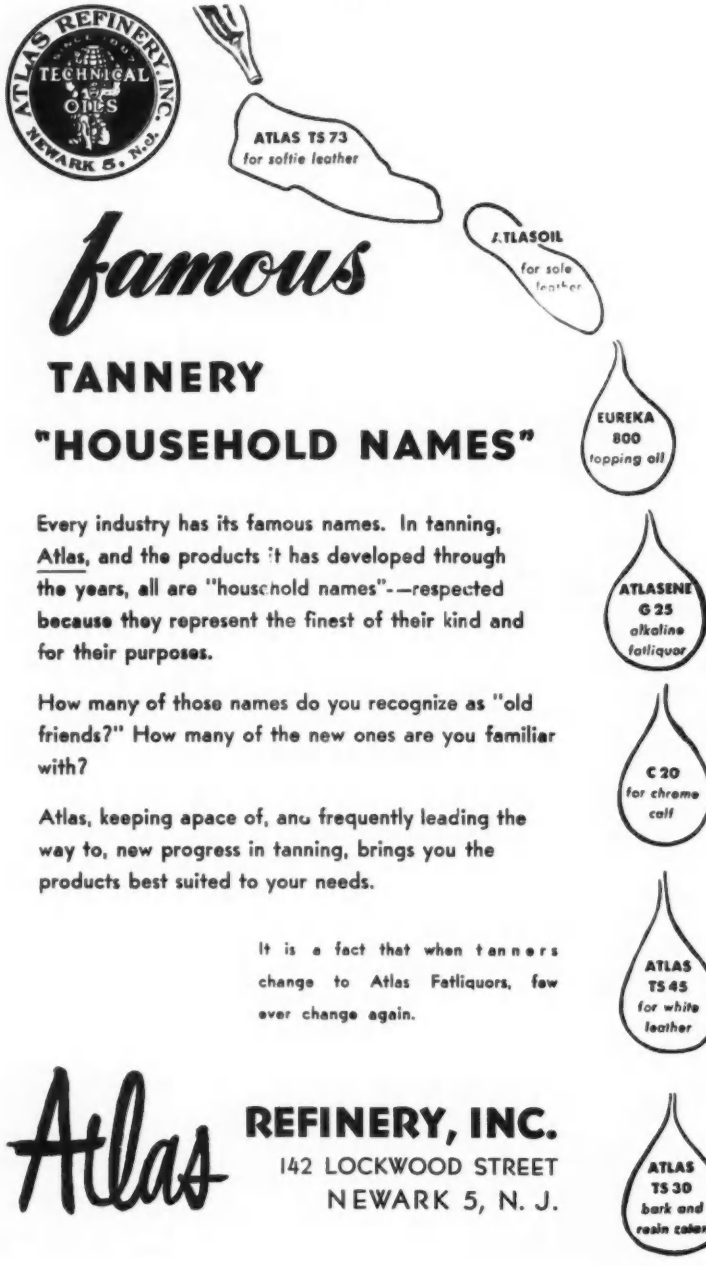

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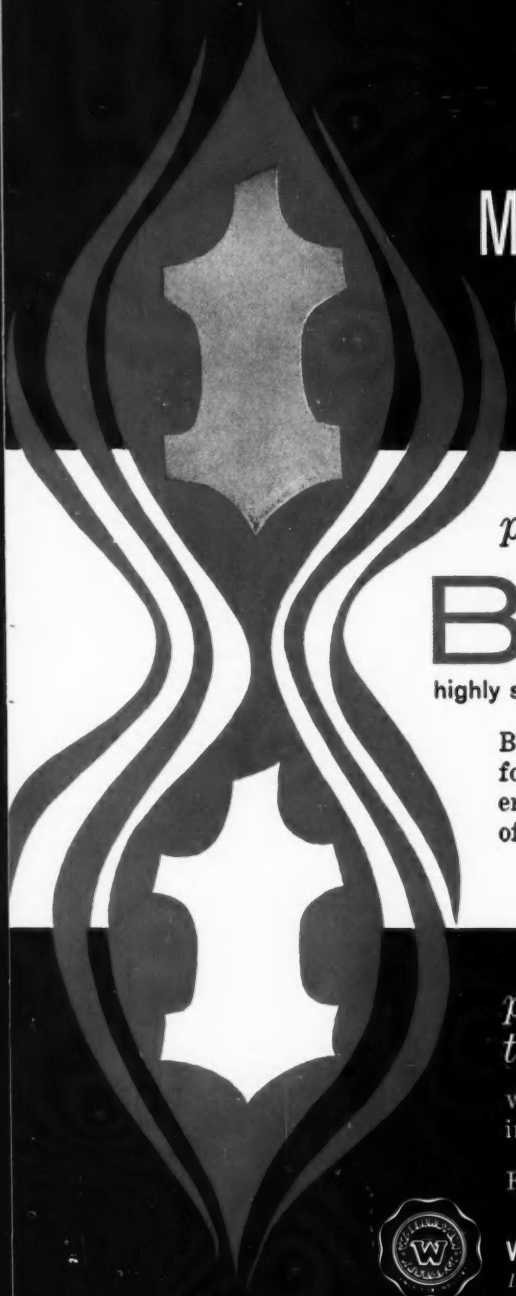

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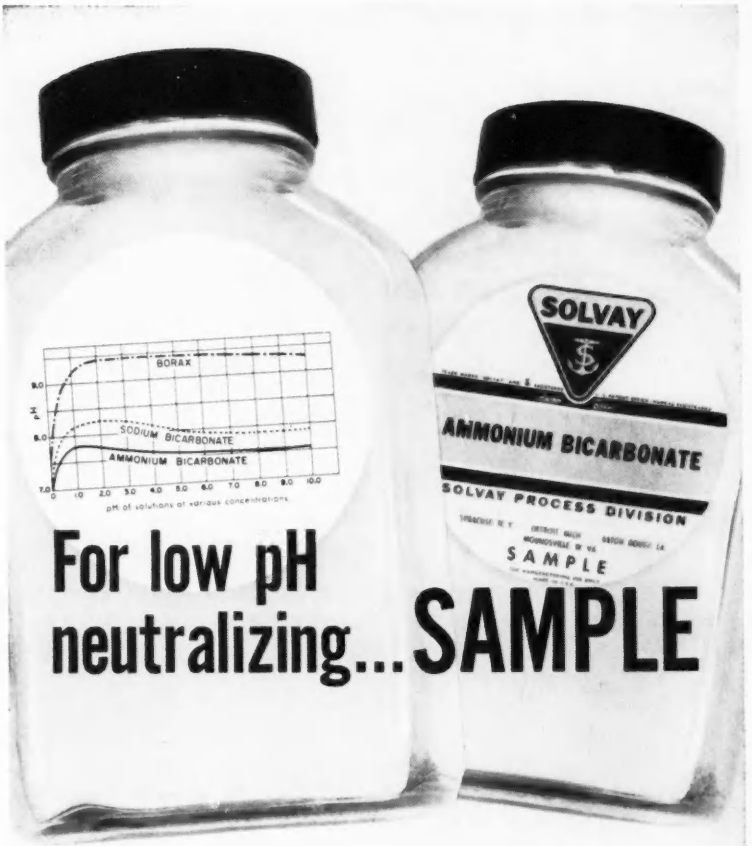

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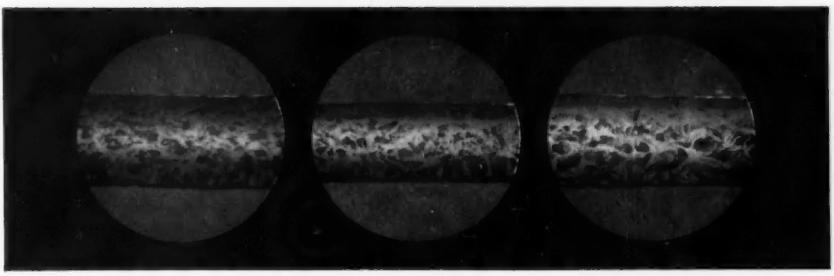

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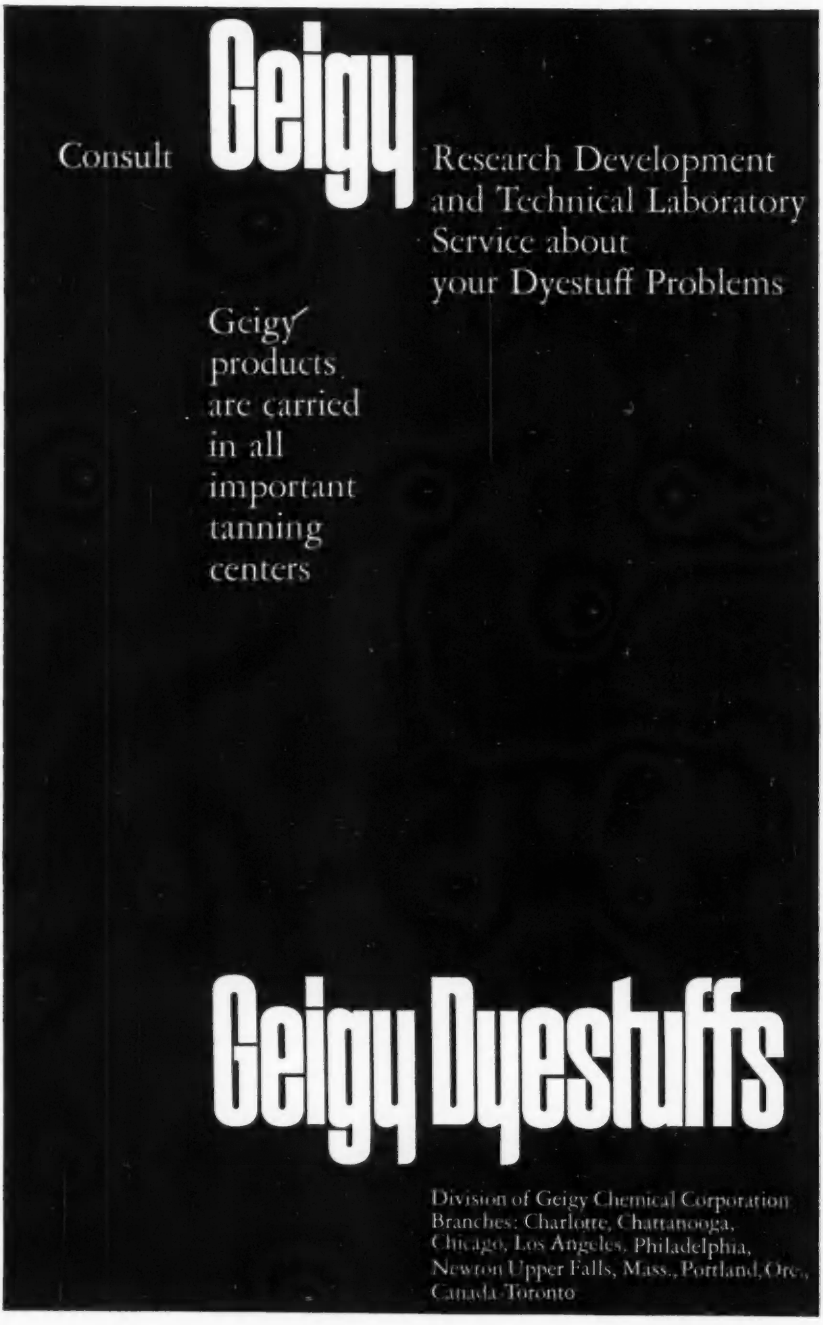
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THE INFLUENCE OF TEST METHODS UPON THE SHRINKAGE TEMPERATURE OF IMPREGNATED SOLE LEATHER

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ABSTRACT

Interest in shrinkage temperature properties of resin-rubber-impregnated sole leather has been aroused by the use of shrinkage temperature measurements as a part of the quality control of such leather. The standard method requires shrinkage temperature measurements on chloroform-extracted specimens in a glycerin-water mixture. A number of commercially impregnated leathers were obtained in order to compare the use of glycerin solution and of water and to study the effect of prewetting on shrinkage temperature measurements. The data presented illustrate the advantages of presoaking in water at 25°C. and also of testing for shrinkage in water as the heating medium. When this procedure is followed, the shrinkage temperature measurements at once become reproducible and meaningful in the sense that they reflect actual resistance to wet heat. In glycerin solutions the shrinkage temperature of vegetable sole leather, whether or not it is impregnated, is lower than in water. Extraction with chloroform also lowers the shrinkage temperature of impregnated vegetable sole leather, but it raises the shrinkage temperature of impregnated chrome retan sole leather. These findings are discussed in the light of related observations reported in the literature.

INTRODUCTION

The resistance of wet leather to heat shrinkage, abbreviated T_s, is one of the most important criteria of tanning. No leather laboratory can operate effectively today without a device for measuring it. Therefore, T_s requirements are gaining wider use as a means for controlling quality standards in the procurement of leather and are, indeed, frequently found in Federal and Military Specifications.

In the past, the practice of setting up T_s requirements was limited to mineral leather. Recently T_s requirements were also included in provisional specifications covering the procurement of resin-rubber-impregnated sole leather of the type developed by the National Bureau of Standards (1, 2). These T_s measurements were to be conducted in accordance with the Federal Specification method, except that the specimens were to be chloroform-extracted.

A T_s determination appears useful as a reassurance against a slack tannage which could be covered up by the impregnation process, or against heat damage caused by a hot-dip or by exceeding safe limits of temperature in expelling the petroleum solvents from the treated leather. However, no data on such leather had been published prior to the issuance of these procurement requirements. A necessity was felt for providing such data and for expanding them in order that the thermal-shrinkage properties of impregnated leather could be fully understood.

In scientific literature the shrinkage resistance of leather is often referred to as the "hydrothermal stability" (3, 4). As this term implies, the shrinkage test is aimed at measuring the shrinkage of a *thoroughly wetted specimen* in water, in contradistinction to a method of measuring the heat shrinkage of a *dry* piece of leather or of *dry* fibers. In most instances wetting occurs within seconds after the specimen is immersed in the bath or in the early stages of heating the bath to the shrinkage temperature. Sole leather, because of its compact nature, requires much more time to wet through. Therefore, specimens frequently do not become thoroughly wet on the inside while being heated for the determination of the T_s by the official method. As a consequence, they fail to shrink even after being left for considerable time at a temperature far exceeding the range characteristic for the T_s of vegetable leather. Since impregnants lower the water absorption, they aggravate this condition, but they do not cause it. Hence, their removal by chloroform extraction only solves part of the difficulty. It is the object of this investigation to explore the problem of how to determine the hydrothermal stability of impregnated sole leather and to suggest such changes as are necessary for consideration in setting up future specification requirements.

EXPERIMENTAL

Samples of two types of impregnated sole leather were obtained in accordance with the Interim Specification mentioned above, including vegetable sole leather from four different sources and chrome-retanned sole leather from one tannery. In Table I these samples are identified by the leathers A to E, respectively.

A Theis shrinkage meter was used for most T_s measurements. The bath was filled either with water or with the glycerin-water mixture required by

TABLE I

WET SHRINKAGE OF IMPREGNATED SOLE LEATHER

Tannage	Tannery	Heating Medium	Degrees Centigrade				
			a	*b*	*c*	*a-c*	*b-c*
Vegetable	A	water	*	83	82	>18	1
		glycerin	*	80	76	>40	4
	B	water	85	86	84	1	2
		glycerin	*	80	77	>39	3
	C	water	82	86	83	−1	3
		glycerin	102	81	77	25	4
	D	water	80	77	72	8	5
		glycerin	83	76	71	12	5
Chrome	E	water	116	110	115	1	−5
		glycerin	109	104	107	2	−3

* = no shrinkage
a = chloroform-extracted, not presoaked
b = not chloroform-extracted, presoaked
c = chloroform-extracted, presoaked

the official method 7011 of Federal Specification KK-L-311a, which from now on will be called "glycerin solution." A transverse shrinkage meter (5) was used for testing the chrome-retanned leather in water. The specimens were tested under the following conditions: (*a*) chloroform-extracted, not presoaked; (*b*) not chloroform-extracted, presoaked; (*c*) chloroform-extracted, presoaked; (*d*) air-dry, no prior treatment.

All T_s figures quoted later represent means of duplicate or triplicate measurements deviating no more than 1°–2°C. from each other. The medium for presoaking was water regardless of whether the heating medium was water or glycerin solution. Each specimen was presoaked separately in a test tube in 20–25 ml. water of about 25°C. for 16–20 hours. The length of time necessary for soaking was established in a series of tests in which the T_s was determined in duplicate after 4, 8, 16, and 24 hours of immersion.

Specimens soaked 4 hours shrunk generally, and specimens soaked 8 hours occasionally, higher than specimens soaked 16 hours. No significant differences between specimens of the same type of leather wetted 16–24 hours were found.

RESULTS

The results of the T_s measurements, except for those obtained in the air-dry state (conditioned) are presented in Table I. The observations under air-dry condition may be summed up as follows:

Impregnated vegetable leather, with one exception, failed to shrink before the water or glycerin solution began to boil. Impregnated chrome-retan leather did not shrink in boiling water but shrunk in the glycerin solution at 105°C. Evidently, wetting of air-dry sole leather proceeds too slowly in most cases to allow shrinkage to occur before the bath has reached the boiling stage.

Table I shows that the method now required as standard (condition *a*) in most cases accelerates rewetting so that T_s measurements can be made; however, a regular procedure for rewetting (conditions *b* and *c*) is more efficient as the generally lower T_s figures indicate. The main conclusion is that the T_s results depend as much on the way the specimens have been prepared for the test as on the heating medium.

Table I shows, for example, that the T_s values of impregnated vegetable sole leather are lower in glycerin than in water after presoaking whether the specimens have been chloroform-extracted or not, and that the T_s values are lowest in both media after the combination of presoaking and extracting.

Although only one chrome sole leather was tested, it is apparent that this leather, too, gave *higher* T_s measurements in water than in glycerin solution. In both media the T_s values were highest after chloroform extraction, lowest after presoaking, and somewhere between these extremes after the combination of extracting and presoaking.

DISCUSSION

The importance of prewetting—previously stressed for conventional sole leather (6)—has been confirmed for T_s studies on impregnated leather. In this special case, presoaking cannot be replaced by extracting with chloroform. More evidence has also been provided to show to what extent T_s measurements in water and in a glycerin solution differ.

Originally, glycerin was proposed (7) solely because it extended the usefulness of a conventional shrinkage meter by about 16°C. At that time, no study was undertaken to reconcile T_s values obtained in glycerin solutions with those found in water. In opposing the use of glycerin, Gustavson (8*a*) pointed to earlier findings that the lower the dielectric constant of the heating medium, the higher is the T_s of collagen, and that because of this relationship, collagen as well as leather has a higher T_s in glycerin solutions than in water (8*b*, 9, 10). Corroborating evidence was furnished recently for aldehyde- and chrome-tanned leather which were found to shrink in glycerin solution 7°–8°C. and 12°–27°C., respectively, higher than in water (6, 11). At the same time only one type of vegetable leather was examined which shrunk in glycerin solution 2°C. *higher* than in water (6). More data evidently were needed. Three more brands of vegetable sole leather were, therefore, compared as to their T_s in the two media; their T_s in glycerin solution was 2°–

6°C. *lower* than in water. Hence, the sole observation previously reported (6) constitutes an exception. In general, glycerin depresses the T_s of vegetable leather whether or not it is impregnated, while it has the opposite effect on aldehyde and chrome leather. Impregnated chrome sole leather resembles vegetable leather in giving lower T_s values in glycerin solution than in water. It is suggested that the impregnation in this case acts like a diluent, as does vegetable tannin, causing the T_s to decline. Chrome and aldehydes, on the other hand, forming covalent cross links, cannot act as diluents as shown recently by Witnauer and Fee (12). Leather tanned by these tanning agents, therefore, behaves like native hide when heated in solutions of different dielectric constants.

Objection to the use of glycerin solution for T_s measurements on sole leather do not have to be based on theoretical grounds alone. The sole practical purpose for employing glycerin, i.e., that T_s values would exceed 100°C., does not apply to any kind of sole leather, including chrome or impregnated, since the highest T_s required by the current specification is only 90°C. An additional difficulty arises from the fact that the rewetting logically should be done in the same medium as the T_s determination. Since a 75% glycerin solution is about 30 times more viscous than water at 25°C. (13), it is obvious that rewetting in glycerin solution would proceed very slowly and not as uniformly as in water.

Once the question of using the same medium for rewetting and for heating the specimens is ettled in favor of water, the only point still to be considered is whether a chloroform extraction is also required. The final column of Table I shows that after extracting with chloroform, the T_s of presoaked vegetable leather in water is lower, and the T_s of presoaked chrome-retan leather higher, than without extraction. The greatest decrease (5°C.) was found on leather D which exhibits the lowest shrinkage resistance of all specimens tested. When the extraction with chloroform is conducted in a Soxhlet apparatus the leather is heated for 7–8 hours to 53°C. It was of interest to ascertain whether the heating itself could be a factor in reducing the T_s of chloroform-extracted specimens. A number of them, therefore, were repeatedly shaken out in the cold with chloroform and then presoaked. These specimens, representing A, B, and C in Table I, had the same T_s in water and in glycerin solution as others cut out next to them that had been extracted in a Soxhlet apparatus. A third series of specimens soaked in water of 53°C. for 8 hours before being tested gave the same T_s as after soaking 16–22 hours at room temperature. Hence, the observation that a prior ex-extraction of chloroform lowers the T_s of presoaked specimens cannot be attributed to prolonged heating.

A specimen of sole leather containing impregnant and one extracted with chloroform were soaked in water. Thermocouples were fixed on the surface and in the center equidistant from the four large surfaces of the pieces. In

separate tests they were then mounted in the Theis shrinkage meter. With the aid of a Brown Automatic Temperature Recorder the temperature gradients between the surface and the center of the specimens during heating and and at the moment of shrinkage were observed. The T_s values for leather C in water as the heating medium in °C. were as follows:

	Not Extracted	Extracted
On the Surface	85.8°	77.2°
At Center	79.8°	71.1°
Difference	6.0°	6.1°

Similar differences were observed on other leathers. In glycerin solution slightly higher temperature gradients (8.5°–9.5°C.) were obtained. In no case was the lag between the surface and the center of the specimens significantly smaller after they had been pre-extracted. Hence, a more rapid transmission of heat into the inner layers in the case of extracted specimens is not the cause of their lower T_s.

In the light of the evidence presented, it appears that the extraction step lowers the T_s of impregnated leather because it restores, in the words of Witnauer and Fee (12), "the availability of the polypeptide chain to the shrinkage medium." While these authors considered only tanning as an impairment of solvation, it is quite apparent that a hydrophobic resin-rubber coating of the individual fibers can function in a similar manner.

Chrome-retan leather, according to Table I, has a higher T_s after the chloroform extraction than before. The most probable explanation seems to be that exposure to heat during the extraction favors the formation of more stable chrome complexes. Gustavson has shown that gradual heating of leather tanned by unstable noncationic chrome complexes is sufficient to elevate its T_s because more stable and more heat-resistant cationic complexes are forming (8*c*; 14*a*, *b*, *c*).

CONCLUSIONS

The results of this investigation are interpreted as strongly favoring the following modifications:

(*a*) the specimens should be thoroughly wetted in water,

(*b*) water should be used as the heating medium, and

(*c*) the chloroform extraction should be conducted in a Soxhlet apparatus according to method 6321 of Federal Specification KK-L-311a.

In order for these conditions to be observed in future purchase descriptions and specifications concerning impregnated sole leather it seems desirable to spell directions out in greater detail. If this is done, military and civilian procurements would be tested under well defined conditions which are a prerequisite for obtaining interlaboratory agreement.

The present study is being extended by a study of storage effects in climatic chambers, since drastic changes have been noted in other types of leather within a comparatively short time of storage (15).

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We wish to thank Messrs. R. Myers and E. C. Dogliotti for their valuable assistance in this investigation.

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AUTOMATIC END POINT DETECTION FOR WATER-PENETRATION TEST E31 WITH LEATHERS TREATED WITH THREE REPELLENT SYSTEMS

REPORT OF THE ALCA WATER PENETRATION SUBCOMMITTEE

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ABSTRACT

Three end point detection methods have been compared for water-penetration test E31, using three different water barrier systems, each at three different levels. The results have been statistically interpreted in accordance with appropriate tests of significance. It is recommended that the visual end point detection method presently used be replaced with an automatic end point detection method (in which the trough made by the test specimen in the Maeser machine is filled with moist machinists' waste and weighted with a length of roller chain) to provide for a more drastic test with electronic end point detection. Data are also presented which show the precision of the method where all three parameters of the test method are considered. It is recommended that greater consideration be given to water absorption of the leather at 10,000 cycles as a method of characterization of water barrier systems instead of placing entire emphasis upon the cycles for initial penetration. The two are shown to be inversely related on a semilogarithmic basis, and water absorption measurements are more precise and more readily determined, especially upon resistant leathers.

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INTRODUCTION

Maeser and Lollar (7) published in 1956 a comparison of three end point detection methods for the Association's water-penetration test E31; this comparison was made upon a side leather which had been treated by a silicone material to give two different levels of increased water resistance to the

particular side leather chosen. That article described the two automatic end point detection methods which were compared to the visual end point detection method now included in the method. Data were also presented with respect to the significance of the results secured. The article also noted the extreme variability of cycles for initial penetration and suggested that this may be an unrealistic parameter for use in measuring the effectiveness of a water barrier system.

These original data were secured upon 5½-oz. chrome-tanned side leather which had been made resistant to water penetration by treatment with Sylflex (2). We have now extended the same experiment to compare the end point detection methods on two more water barrier systems applied to the same type and weight of leather. The first of the new treatments has been given the trade name of Bavon (5), and its properties have been described by von Fuchs (9). The second of these has been produced by the Niagara Chemical Company, and this process has been developed by Hopton (4). The characteristics of all three systems are reviewed by Dudley (3). The leather for each of the three treatments was secured from a different source. The treating was done, in each case, under the direction of the manufacturer of the waterproofing material. The sampling plan used for the Bavon and the Niagara Chemical Company treatments was the same as that described for the Sylflex treatments in the original paper. The data were secured and presented to characterize the test method but not the methods of treatment. Different treatments were used to secure different response levels so that the test method could be characterized more effectively. The results are not interpreted by the authors to be representative of the response of any one of the three treatment methods when carried out on lots of leather under conditions defined by those who produce the several barrier systems which were used.

THE PRECISION OF THE RESULTS

Method E31 provides that three different indicators of the water barrier characteristics of a specimen shall be noted to characterize it; these three are water penetration, water transmission, and water absorption. Data were secured on all three of these characteristics in this work, and wherever possible the results secured were examined by the appropriate statistical techniques contemplated in the design of the experiments to estimate the precision of the results secured from use of the method.

The experimental design did not attempt to make intercomparison of the results secured from the different water barrier systems or from the different levels of a given barrier system. This design was restricted to an examination of the effect of the three methods of end point detection upon the results. For each of the three water barrier systems at each of the three levels, the following Latin cube experimental design (1) was applicable:

Source of Variance	*Degrees of Freedom*
Between end point methods	$3-1=2$
Between sides of leather	$3-1=2$
Between rows	$3-1=2$
Between columns	$3-1=2$
Error	18
Total	$3\times3\times3-1=26$

This Latin cube design was used so that the known large variation of the water barrier characteristics between and within sides could be estimated so that the assessment of the effect of the end point methods could be made with maximum efficiency. Also, the error variance of course permits one to make an estimate of the inherent error of the results secured, and this estimate is shown in the tables which follow.

Tables I, II, and III summarize data that were secured in the nine different analyses of variance conducted in accordance with the design planned for the experiment. Because of the nature of the data being secured, it was not always possible to carry the examination of a given specimen to the chosen end point. Since the use of a Latin cube design imposes some rigorous limitations upon the type of data which can be subjected to analysis of variance, it was not always possible to apply the contemplated analysis of variance to the data secured. In this case an appropriate non-parametric ranking test of significance (10) was applied to the data secured so that a comparison could be made of the effect of the three different end point detection methods upon the results. All statistical decisions were made at the 5% level of significance; those decisions whose probability level lies between the 10% and the 5% level have been indicated with a question mark.

Each of the three tables presents data for all nine of the combinations of water barrier system and level. Table I presents the data for the cycles for initial penetration of the specimen, Table II presents data upon the water transmitted by the specimen at 10,000 cycles (with the transmission reported in grams), and Table III presents data on the water absorbed by the specimen at 10,000 cycles (again the data are in grams). The first three data columns in each table present the average value secured by each method of end point detection. The last four columns report the results from the application of the appropriate statistical methods to the data. The standard deviation and the coefficient of variation (100 × standard deviation/average) have been estimated from the error term in the analyses of variance. Since some of the specimens could not be carried to the contemplated end point, it was impossible to secure data for these precision estimates in several cases. These cases are indicated in the tables by a + symbol after the average value, since the completion of the tests on these incomplete specimens would have in-

TABLE I

A COMPARISON OF THE RESULTS SECURED FOR CYCLES FOR INITIAL PENETRATION BY THE THREE END POINT METHODS

Treatment Method and Level	Average Number of Cycles			Statistical Parameters			
	End Point Method A	End Point Method B	End Point Method C	Grand Average	Standard Deviation	Coefficient of Variation %	Method Significance
Niagara Control	130	174	94	133	27	20.3	Yes
Bavon Control	398	557	440	465	226	48.6	No
Sylflex Control	498	398	448	448	116	25.9	No
Niagara Intermediate	4259	2175	1099	2511	1727	68.6	Yes
Bavon Intermediate	195	180	190	188	88	46.7	No
Sylflex Intermediate	15540	10700	8680	11640	6050	52.0	?
Niagara Maximum	8402	6537	3209	6049	3849	63.6	Yes
Bavon Maximum	9939+	5206	3021	6055+	Indeterminant+		Yes
Sylflex Maximum	40000+	22620	14940	25850+	Indeterminant+		Yes

+Certain specimens were terminated before detection of penetration, hence precision estimates are indeterminant.

TABLE II

WATER TRANSMISSION AT 10,000 CYCLES FOR THREE END POINT METHODS

Treatment Method and Level	Average Number of Grams			Statistical Parameters			
	End Point Method A	End Point Method B	End Point Method C	Grand Average	Standard Deviation	Coefficient of Variation %	Method Significance
Niagara Control	10.97	11.83	10.42	11.07	2.14	19.3	No
Bavon Control	6.47	6.99	6.93	6.80	1.59	23.4	No
Sylflex Control	8.58	6.32	4.42	6.44	2.13	33.1	Yes
Bavon Intermediate	6.02	8.20	10.5+	8.3+		Indeterminant	Yes

Niagara Intermediate, Sylflex Intermediate, and all three agents at maximum level showed no significant water transmission.

TABLE III

A COMPARISON OF THE RESULTS SECURED FOR GRAMS OF WATER ABSORBED BY SPECIMEN WITH THE THREE END POINT METHODS AT 10,000 CYCLES

Treatment Method and Level	Average Number of Grams			Statistical Parameters			
	End Point Method A	End Point Method B	End Point Method C	Grand Average	Standard Deviation	Coefficient of Variation %	Method Significance
Niagara Control	8.47	8.24	7.92	8.21	0.63	7.3	No
Bavon Control	6.43	7.08	7.40	6.97	0.96	13.8	No
Sylflex Control	3.92	4.27	3.73	3.98	0.30	7.5	Yes
Niagara Intermediate	3.06	3.37	3.62	3.35	0.81	24.2	No
Bavon Intermediate	9.18	10.82	10.86+	10.3+		Indeterminant	Yes
Sylflex Intermediate	0.63	1.17	1.57	1.12	0.24	21.0	Yes
Niagara Maximum	1.46	2.50	2.93	2.30	1.11	48.5	Yes
Bavon Maximum	1.85	2.80	3.15	2.60	0.89	34.0	Yes
Sylflex Maximum	0.47	0.77	1.02	0.75	0.11	13.3	Yes

creased the average values. Furthermore, the standard deviation could not be calculated because of this truncation of the testing; hence, the data for precision are expressed as "indeterminant". However, the non-parametric ranking techniques did make it possible to draw valid conclusions about the comparison of the methods even in these truncated sets of data; hence, the final column of the table headed "Method Significance" does have an entry in every case.

Inspection of the results secured will make it evident that the three methods of end point detection do not give identical results in many of the different sets of leathers. Methods B and C were the electronic automatic end point detection methods; hence, these methods would be preferred because of the increased ease with which the testing could be conducted. Method B has the trough of the specimen filled with moist machinists' waste during the test, while Method C is identical to Method B except that the waste is held in close contact with the specimen by the weight of two lengths of roller chain as described earlier (7). It will be noted that Method C is more severe on the leather, causing penetration of the treated leathers in fewer cycles and increasing the amount of water absorbed by the specimen in 10,000 cycles. This method of end point detection was preferred in the previous partial summary of this work, and the data presented here merely strengthen the conclusion given earlier that Method C should be adopted to replace the visual end point detection method.

Comparison of the statistical parameters in Tables I, II, and III has demonstrated the very great variability in the cycles for penetration of water-resistant leathers. This has been characteristic of all attempts to establish precision estimates on this parameter. While the water transmission data are very incomplete, it may be noted that the water absorbed by the specimen is generally somewhat more concordant, as will be seen from an examination of the columns that are second from the right in Tables I and III.

Graphical inspection of the data for cycles for initial penetration and the amount of water absorbed by the specimen at 10,000 cycles suggested that the two factors were inversely related logarithmically. Application of the appropriate statistical techniques of analysis of regression (8) to the data secured from the three different water barrier systems showed that this was in fact true. When the common logarithm was taken of the cycles to secure penetration, and when this logarithmic value was related to the grams of water absorbed at 10,000 cycles, the following interrelationships were found:

	Coefficient of Correlation r	*Coefficient of Covariation* $r2$
Niagara System	−0.8872	0.7871
Bavon System	−0.8125	0.6602
Silicone System	−0.9494	0.9014

Application of the technique of analysis of regression to the data derived the following equations, if the assumption is made that all of the error is to be found in the cycles to penetration:

Niagara System	$Y_r = 4.06 - 0.237\ X$
Bavon System	$Y_r = 3.78 - 0.148\ X$
Silicone System	$Y_r = 4.54 - 0.468\ X$

In each of these equations, Y_r is the logarithm of the cycles to produce initial penetration; it should be recalled that the logarithm of 10,000 is 4. X is the grams of water absorbed by the specimen in 10,000 cycles. These three equations were found graphically to represent quite well the scatter diagram resulting from the plotting of the original data on semilogarithm paper. Analyses of regression of the data secured also showed that the inverse linear relationship was quite significant, since the 0.1% probability level was exceeded in all three cases.

This relationship is emphasized to illustrate the desirability of giving greater consideration to the other two end point parameters of water-penetration test E31 as well as cycles for initial water penetration. Since the amount of water absorbed by the specimen at 10,000 cycles is inversely related to the cycles to penetrate, and since it seems in general to be capable of more precise determination, it could be that an improved rating of the water barrier characteristics of different leathers would result from the use of amount of water absorbed at 10,000 cycles as the primary quality index. In addition to the greater precision which we have noted, the indicator would also have the advantage that the test could be terminated in a more reasonable time. With the Maeser tester being operated at 200 rpm., this end point would be reached in 50 minutes; this time makes the method much more readily applicable than it is when it must be carried to an indefinite end point requiring several hours.

It must be realized that the final value of either end point parameter is dependent upon its correlation with the response of the leathers in actual wear. It may be noted that the maximum ratio between treated and untreated leather for end point detection Method C found in the data in Table I where cycles to penetrate are considered is 14,940/94 or approximately 150. On the other hand, these same two sets of leather show a ratio of 7.92/1.02, or approximately 8, when the data in Table III are considered. It is, of course, unknown as well as difficult to estimate what the actual service utility of these two blocks of leather would have been if they had been placed into shoes. It should be emphasized at this point that the original calibration of the Maeser test method against actual wear conditions (6) was made upon leathers which did not show the extremely high levels of cycles for initial penetration.

RECOMMENDATIONS FOR ACTION ON METHOD

It is recommended that the following action be taken by the ALCA-ASTM Joint Committee on Leather:

(*a*) The description of the water-penetration test E31 now included in the Methods Booklet of the Association should be amended to include a description of the automatic electronic end point detection which is coded C in this report to lessen the tedious attention which is necessary when the present visual end point detection method for initial penetration is used.

(*b*) This report, together with earlier work which is referenced in this paper, should be referred to the water resistance section of the Joint Committee on Leather for their use in the development of the desired discussion on significance and precision of the test method results which the Joint Committee has requested in the revised method descriptions. It is recommended that particular attention be given to the precision estimates included in the report and to the suggestion that more attention be given to the water absorption of the specimen as a parameter in the description of a water-resistant leather.

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Received February 19, 1959.

There are more than 12,500 chemical-producing plants in the U.S. More than $1 billion has been spent each year since 1951 on new facilities and the annual average for the past 14 years exceeds $1 billion. Chemical plants costing an estimated $1.5 billion and involving 304 projects were under construction or scheduled for completed construction in the 1959-60 period.

—*The Chemical Industry Facts Book*, 1960-61 *Edition*. Manufacturing Chemists' Association, Inc., Washington, D. C

STRENGTH AT GRAIN CRACK OF CALFSKIN LEATHERS

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ABSTRACT

An analysis of the values of strength at grain crack obtained on 179 calfskin leathers indicates that the measurement gives values which fit a model normal distribution. At a minimum requirement of 300 lb. at grain crack approximately 30% of the specimens would fall below the requirement.

INTRODUCTION

Recently in a study of the quality of calfskin leathers (1) sufficient skins were available to obtain a distribution curve of the strength values at grain crack of the leathers. The Mullen strength value at grain crack has been used by both tanner and shoe manufacturer to check lots of leather. The present report indicates the limits which may be placed on the strength at grain crack of calfskin leathers.

In the previous study (1) the calfskins were purchased to determine if any difference existed between the cold-pull and the hot-flay practices of handling calfskins. The results obtained indicated that no difference in tensile strength existed which could be attributed to the cure practices. When the present investigation was undertaken, 179 of the 240 calfskin leathers were available for test. Approximately one half of the calfskin leathers were made from calfskins removed from the carcasses by the hot-flay practice, and the other half were made from calfskins from carcasses where the skins were removed by the cold-pull practice.

Sixty of the calfskins (30 hot-flay calfskins and 30 cold-pull calfskins) were purchased and processed through a tannery in the winter. During the summer 30 calfskins of each practice of removing the skins from the carcasses were purchased and processed through the same tannery. An additional 30 calfskins of each practice were purchased and processed into leather in a second tannery during the summer.

The calfskins were processed at each tannery with the regular stock through chrome tannage and were finished as tan aniline leathers. For identification the calfskins and the leather were numbered from 1 to 180. One of the skins was lost in process to give a total of 179 calfskin leathers.

Based on visual inspection, microstructure, chemical analyses, and tensile strength of the salt-cured stock and the finished leather, there were no detectable differences in the cold-pull skins versus the regular-flay skins. Therefore it was thought that the 179 calfskins would give a fair representation of grain crack strength of calfskin leathers.

TESTING PROCEDURE

From each of 179 calfskin leathers a 3″ x 3″ block was cut from the area shown in Fig. 1. The specimen was located 6″ in from the root of the tail and 1″ in from the backbone.

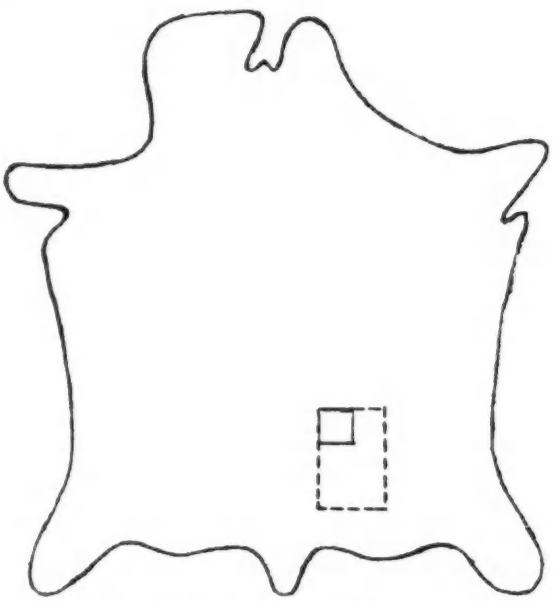

FIGURE 1.—The specimen is located 6″ from the root of the tail and 1″ from the backbone.

The specimens were conditioned at 50% ± 5% relative humidity and 72° ± 1.8° F. for 48 hours before testing for grain crack strength. After the specimens were conditioned, they were tested on a hand-driven Mullen tester until grain cracking appeared. The values obtained are given in Table I.

TABLE I

STRENGTH AT GRAIN CRACK OF CALFSKIN LEATHERS

Skin	Thickness Inch	Lb. at Grain Crack	Skin	Thickness Inch	Lb. at Grain Crack	Skin	Thickness Inch	Lb. at Grain Crack
1	.047	350	43	.036	160	85	.046	470
2	.067	520	44	.031	210	86	.065	330
3	.050	380	45	.038	230	87	.043	440
4	.050	290	46	.064	160	88	.040	410
5	.071	270	47	.071	270	89	.052	800
6	.061	460	48	.041	270	90	.046	570
7	.065	430	49	.056	340	91	.047	380
8	.056	600	50	.064	410	92	.045	380
9	.041	190	51	.058	550	93	.055	430
10	.061	260	52	.058	450	94	.051	520
11	.063	460	53	.066	510	95	.051	480
12	.064	240	54	.071	220	96	.052	330
13	.044	220	55	.064	510	97	.043	480
14	.040	320	56	.061	300	98	.050	330
15	.050	370	57	.057	270	99	.049	170
16	.062	370	58	.053	290	100	.037	530
17	.042	180	59	.069	420	101	.065	280
18	.053	500	60	.058	360	102	.053	270
19	.045	220	61	.060	200	103	.063	340
20	.051	450	62	.043	290	104	.043	330
21	.039	190	63	.058	220	105	.062	260
22	.056	380	64	.051	260	106	.043	520
23	.057	290	65	.050	290	107	.043	520
24	.031	300	66	.053	410	108	.038	490
25	.035	220	67	.068	640	109	.056	420
26	.038	270	68	.055	400	110	.046	590
27	.060	370	69	.053	170	111	.059	510
28	.061	320	70	.054	450	112	.052	590
29	.065	470	71	.062	330	113	.054	360
30	.060	510	72	.056	320	114	.045	400
31	.067	280	73	.065	460	115	.047	290
32	.068	330	74	.051	330	116	.053	730
33	.058	280	75	.073	160	117	.044	280
34	.036	250	76	.048	370	118	.047	270
35	.038	280	77	.048	330	119	.052	330
36	.033	180	78	.046	560	120	.043	358
37	.032	220	79	.056	400	121	.042	210
38	—	—	80	.041	320	122	.056	370
39	.033	340	81	.042	280	123	.037	380
40	.036	250	82	.041	380	124	.042	380
41	.038	210	83	.048	280	125	.041	290
42	.041	340	84	.046	540	126	.053	470

TABLE I - *Continued*

Skin	Thickness Inch	Lb. at Grain Crack	Skin	Thickness Inch	Lb. at Grain Crack	Skin	Thickness Inch	Lb. at Grain Crack
127	.044	320	145	.041	440	163	.046	420
128	.056	420	146	.033	380	164	.056	440
129	.041	210	147	.034	310	165	.051	370
130	.049	480	148	.060	380	166	.032	290
131	.049	600	149	.051	300	167	.043	270
132	.048	430	150	.035	610	168	.053	410
133	.041	290	151	.039	300	169	.034	350
134	.036	320	152	.041	350	170	.040	340
135	.050	370	153	.037	370	171	.040	200
136	.060	400	154	.042	360	172	.047	590
137	.038	350	155	.051	420	173	.035	310
138	.042	480	156	.039	480	174	.033	380
139	.033	310	157	.043	380	175	.055	330
140	.041	410	158	.050	550	176	.034	460
141	.043	260	159	.053	360	177	.058	250
142	.029	340	160	.052	570	178	.058	350
143	.036	310	161	.055	380	179	.039	330
144	.039	330	162	.057	570	180	.053	440

RESULTS

Examination of the data in Table I shows that there is a considerable spread in grain crack strength in each lot of 30 calfskin leathers. To determine if the total data represented a normal distribution, the distribution was determined as given in Fig. 2. The data were then tested for homogeneity by the following methods:

(*a*) Analysis of variance (one factor) for difference between means

(*b*) Bartlett's test for homogeneity of variances

(c) X^2 test for a measure of fit with a normal distribution

The analysis of variance is given in Table II. Examination of the table shows an F value of 1.41. At the 5% level F would have to be 2.27 or higher to be significant. Therefore there is no significant difference shown between the means of the six lots of samples. The parameters of the combined set are then taken to be $\overline{X} = 361.1$ lb.; $\sigma^2 = 13{,}208$ lb.^{2}

The result of Bartlett's test for homogeneity of variances is given in Table III. Examination of the table shows an F value of 1.78. At the 5% level F would have to be 2.21 or higher to show nonhomogeneity. Therefore the variances are homogeneous.

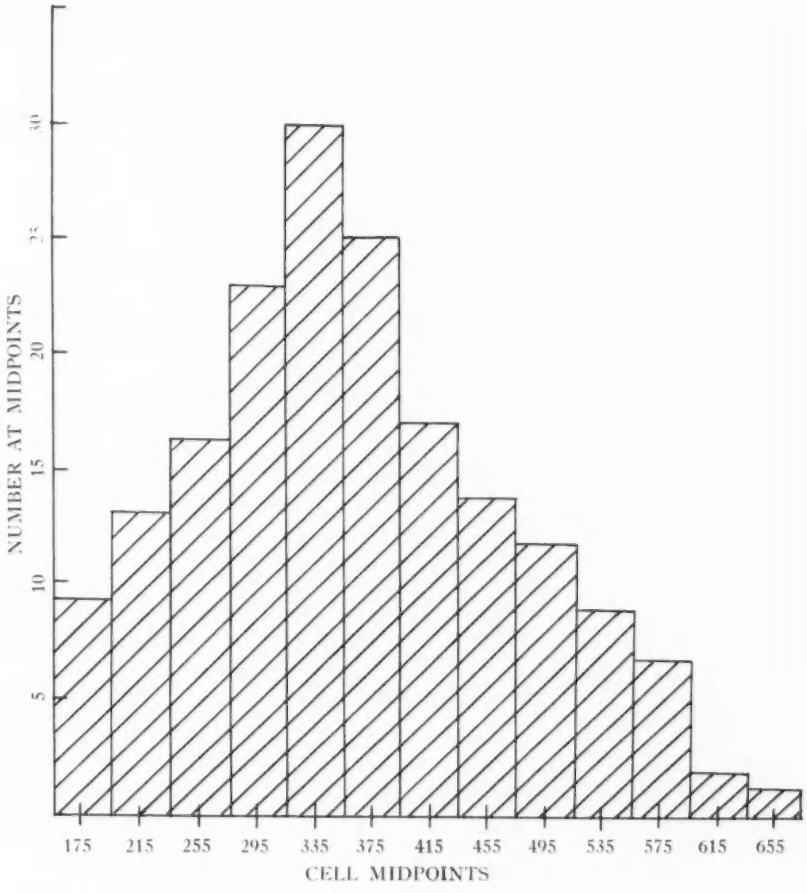

FIGURE 2.—Distribution of grain crack values.

The X^2 test for goodness of fit for normality gave the results in Table IV.

As the critical value of X^2 at the 5% level is 16.9, the value of 7.32 given in Table IV could occur by chance about 60% of the time, even if the samples were taken from a known normal distribution. Therefore, in this case, there is no reason to believe the distribution is not normal.

TABLE II

ANALYSIS OF VARIANCE

(Values are coded by dropping last zero)

LOT	① N	② ΣX	③ ΣX^2	④ $\left(\frac{\Sigma X}{N}\right)^2$	⑤ σ^2
1–30	30	1040	39778	1202	124
31–60	29	889	30295	940	105
61–90	30	1149	49729	1467	191
91–120	30	1216	53788	1643	150
121–150	30	1105	42903	1356	74
151–180	30	1164	47780	1505	88
Total	179	6563	264,273		

Total Variation: $\Sigma_③ - \frac{(\Sigma_②)^2}{\Sigma_①} = 264{,}273 - 240{,}631 = 23{,}642$

Between Sample Variation: $\frac{1040^2 + 889^2 + 1149^2 + 1216^2 + 1105^2 + 1164^2}{30} - \frac{(\Sigma_②)^2}{\Sigma_①}$

ANOVA TABLE

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Between Samples	926	5	186.2	1.41
Residual	22716	173	131.3	
Total	23642	178		

The Critical $F_{.05}(5,173) = 2.27$

TABLE III

BARTLETT'S TEST FOR HOMOGENEITY OF VARIANCE

Lot	σ_i^2	S_i^2	$\ln S_i^2$	$k=6$ $\Sigma n_i = 179$
1–30	124	128.3	4.8543	
31–60	105	108.6	4.6877	
61–90	191	197.6	5.2863	
91–120	150	155.2	5.0447	
121–150	74	76.6	4.3386	
151–180	88	91.0	4.5109	
Total		757.3	28.7225	

$$S_b^2 = \frac{\Sigma S_i^2}{6} = \frac{757.3}{6} = 126.3$$

$$\ln S_b^2 = 4.8387$$

$$M = (N\text{-}k) \ln S_b^2 - \Sigma (ni - 1) \ln S_i^2$$
$$= (173)(4.8387) - 29(28.7225)$$
$$= 841.93 - 832.95$$
$$= 8.98$$

$$A = \frac{1}{3(k\text{-}1)}\left(\frac{k}{ni\text{-}1} - \frac{1}{N\text{-}1}\right)$$
$$= \frac{1}{3.5}\left(\frac{6}{29} - \frac{1}{173}\right) = .0134$$

$$\delta_1 = 5$$

$$\delta_2 = \frac{7}{A^2} = \frac{7}{.00018} = 3889$$

$$b = \frac{\delta_2}{1\text{-}A + \frac{2}{\delta_2}} = \frac{3889}{.9871} + 3940$$

$$F = \frac{\delta_2 . M}{(k\text{-}1)(b\text{-}M)} = \frac{(3889)(8.98)}{5(3940 - 8.98)} = 1.78$$

Critical $F_{.05} = (\delta_1, \delta_2) = 2.21$

TABLE IV

X^2 TEST FOR GOODNESS OF FIT OF NORMALITY

Cell Midpt.	Lower Limit	Corr. Z	Area $-\infty$ to Z	Area in Cell	Calc. Freq. fo	Obs. Freq. Fo	$\frac{(Fo\text{-}fo)^2}{fo}$
175	–	———	———	.0742	13.3	9	1.39
215	195	−1.445	.0742	.0626	11.2	13	.29
255	235	−1.097	.1364	.0907	16.2	16	.00
295	275	− .749	.2273	.1169	20.9	23	.21
335	315	− .401	.3442	.1347	24.1	30	1.44
375	355	− .053	.4789	.1371	24.5	25	.01
415	395	.295	.6160	.1239	22.2	17	1.22
455	435	.643	.7399	.0992	17.8	14	.81
495	475	.991	.8391	.0706	12.6	12	.03
535	515	1.339	.9097	.0445	8.0	8	.00
575	555	1.687	.9542	.0248	4.4	7	1.54
615	595	2.035	.9790	.0210	3.8	5	.38
					—	—	—
					179.0	179	7.32

Critical $X^2_{.05}$ (9 d. f.) = 16.9

The apparent skewness of the distribution is not revealed by the X^2 test because the first cell theoretically is from $-\infty$ to 195, but the actual values were only from 160 to 195. A separate test on skewness revealed a slight degree of skewness, but this is probably due to a truncation in the distribution resulting from a "threshold value" below which the presence of a grain crack cannot be determined in the present Mullen grain crack method.

Any predictions on a minimum requirement for values of the strength at grain crack can be drawn from the model normal distribution, as shown in Fig. 3, with parameters of $\bar{X} = 361.1$ lb. and $\sigma = 114.9$ lb. For example, if a value of 200 lb. at grain crack was chosen as the minimum requirement,

$$Z = \frac{200 - 361.1}{114.9} \quad \frac{-161}{114.9} = -1.40$$

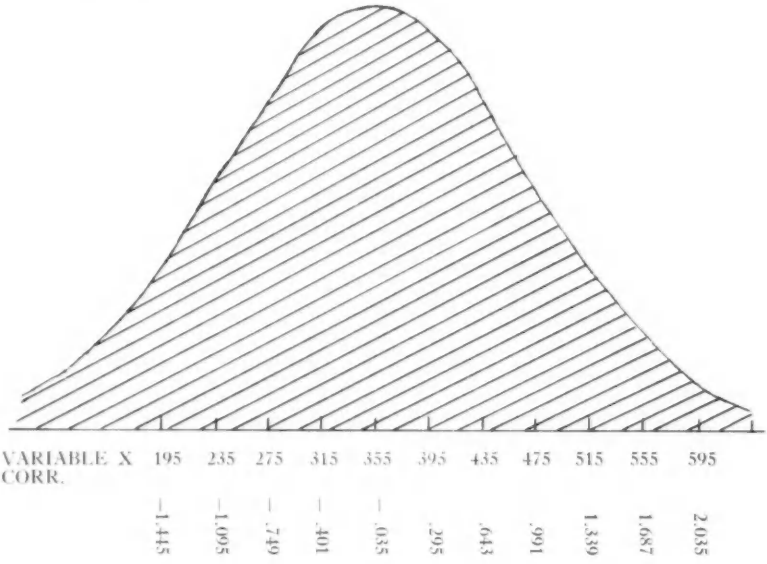

FIGURE 3.—Normal distribution model for grain crack data parameters. $\bar{X} = 361.1$ lb. $\sigma = 114.9$ lb.

From a table of normal areas for a Z value of 1.40 it can be predicted that 8.08% of the specimens will fall below this requirement. If a minimum value of 300 lb. at grain crack is set as a minimum requirement, approximately 30% of the specimens will fall below the requirement. Checking the values in Table I indicated that 29.6% fall below the 300 lb. minimum.

SUMMARY

In the case of the strength at grain crack of calfskin leather the values obtained on 179 different skins indicate that they fit a model normal distribution. Using the value from a table of normal areas it can be predicted that at a minimum requirement of 200 lb. at grain crack 8% of the specimens will fall below the requirements. At a minimum requirement of 300 lb. at grain crack 30% of the specimens will fall below the requirement.

REFERENCE

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A knowledge of the structure of collagen, the leather-forming protein of hides and skins, and of the mechanism of tanning, is of direct industrial importance to the tanners, the shoe manufacturers, and all who are called on to appraise the properties of leather. The Bureau [National Bureau of Standards] recognized the need for more basic information and has directed a large portion of the research work of the Leather Section toward fundamental studies of collagen, concepts of the mechanism of tanning, and measurement of the physical and chemical properties of collagen and leather. The staff of the Leather Section has closely cooperated with the American Leather Chemists' Association as active members, officers on the Council, and members of committees, and it has published more than 80 research papers in the Journal of the American Leather Chemists' Association.

—*Leather Research and Technology at the National Bureau of Standards*, U. S. Department of Commerce. NBS circular 560, June 1, 1955.

An estimated 30.6% of the 79,200 scientists and engineers employed by the chemical industry in January 1957 were engaged in research and development. More than 400 new chemicals or chemical products are placed on the market every year.

—*The Chemical Industry Facts Book*, 1960-61 *Edition*. Manufacturing Chemists' Association, Inc., Washington, D. C.

Until the middle of the 19th century, with transportation facilities limited, chemical manufacturing (in the U.S.) developed to meet local needs. Early demand came from makers of textiles, leather, paper, glass and soap. Philadelphia, strategically located, became the first chemical center.

—*The Chemical Industry Facts Book*, 1960-61 *Edition*. Manufacturing Chemists' Association, Inc., Washington, D. C.

LIFE LINES

Brief Biographies of Our Contributors

HUBERTINA D. CLAYTON was graduated from the University of Philadelphia with a master of science degree in biochemistry after having completed her undergraduate work in biology and chemistry at Seton Hill College, Greensburg, Pa. She has been active in research on tanning and leather development since 1952 in the Leather Technology Laboratory Branch of the Quartermaster Research and Engineering Command.

ROBERT J. FOPMA, A.B., is Associate Professor of Mathematics in the College of Engineering, University of Cincinnati. He has specialized in statistical analyses and served on the Technical Committee of the Upholstery Leather Group. He is also a member of the staff of the Tanners' Council Research Laboratory on a part-time basis.

DR. ROBERT M. LOLLAR became Technical Director of Armour Leather Co. in January of this year. Previous to that time he was an associate professor in the Department of Tanning Research and a member of the staff of the Tanners' Council Research Laboratory, both at the University of Cincinnati where he earned his M.S. and Ph.D. degrees. Dr. Lollar has been a frequent contributor to this *Journal*, and until recently he served as one of its regular abstracters. In 1954 ALCA presented him the Alsop Award.

MIETH MAESER, well known as the President of ALCA, is a graduate of Brigham Young University and Massachusetts Institute of Technology. He has been associated with the United Shoe Machinery Corporation for more than twenty years. Since becoming a member of ALCA in 1941, he has played an important role in devising evaluating, and revising physical test methods for leather.

WILLIAM T. RODDY—See the March, 1960, *Journal*.

DR. LUDWIG SELIGSBERGER, born and educated in Germany, worked both there and in Turkey before coming to the United States in 1937. After many years as chief chemist of the New Castle Specialty Division of Allied Kid Company in Wilmington, Delaware, he joined the Leather Technology Laboratory Branch of the Quartermaster Research and Engineering Center, Natick, Mass., where he is now chief of the Synthetic Tanning Evaluation Section. He has published several papers in this *Journal*.

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J. R. BLOCKEY, M.Sc., F.R.I.C.

In Memoriam

With the death of Mr. J. R. Blockey, which occurred at his home in Corfe Castle, Dorset, England, on February 27, the industry and technology of leather have lost an outstanding personality. His friends and colleagues in many lands feel grief at the loss of an amiable gentleman and a wise counsellor.

Mr. Blockey was born in Leeds in 1886. After completing study for the M.Sc. degree at Leeds University, he joined the Leathersellers' Technical

College in 1909 as one of the original members of its staff. He entered the leather industry in 1913, originally with the intent of gaining practical experience during a few years. However, he came to stay in the industry for some twenty-five years, first as works chemist and later as works manager. Thus, for a long time he was associated with Messrs. Wm. Walker & Co. Ltd. in Bolton. In 1936 Mr. Blockey was appointed Principal of his old college in Bermondsey and remained in that post until his retirement in 1953.

Blockey's influence on the industry is multifarious. As a chemist, he contributed to the knowledge of various processes in tanning, particularly on analytical matters. His paper on the effect of dilution of solutions of chromium sulfates on the pH of the solutions, published in 1918, is in my estimation a very important paper. However, the intrinsic merits of the findings could not be appreciated at the time of its publication, not even by its author, because the relationship of pH and olation was not generally known then. Blockey's paper is one of the very first investigations in applied chemistry with actual measurements of the H-ion concentration As a technologist and practical tanner, he was responsible for important innovations, for instance, in the making of chrome liquors and the application of grease to leather.

As a teacher and administrator, he was respected for his breadth of knowledge, a happy symbiosis of practice and theory rarely found, and for his sincerity, magnanimity, and generosity. Mr. Blockey was also the prime mover in establishing his college as a National College, accomplished in 1951. World War II claimed his services as the Technical Adviser to Leather Control in Great Britain, in which mighty capacity his technical knowledge, practical attitude, and personal integrity proved invaluable. His service to various bodies of the Industry, quietly and unobtrusively, cannot yet be adequately evaluated. His background enabled him to play a prominent role in the early development of leather technology, and to become the first British incumbent of the presidency of the International Union of Leather Chemists' Societies for the period 1949–1951. The 2nd Union Conference in London, held in 1951 under his regentship, is still recalled with gratitude and pleasure.

Mr. Blockey's personal interests outside his profession and intellectual pursuits swing from the frenzy of the football match in his younger days to the tranquillity of the cricket field in later years. Above all, his great fondness for flowers seems to me to be most congruent with his person. He started and finished his day by tending the flowers in his garden and in his greenhouse.

On his usual morning walk on February 27, 1960, after having tended his greenhouse protégés his heart suddenly gave in. It was a farewell appropriate to a devotee of Flora, in Vergers Mead at Corfe Castle in Dorset in Old England. He leaves a blessed memory.

K. H. Gustavson

DIRECTORY CHANGES

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Peter Bedrosian, A. C. Lawrence Leather Company, Calfskin Division, Peabody, Massachusetts.

Philip D. Bixby, Box 223, R.D. No. 1, Endicott, New York. Endicott-Johnson Corporation, Endicott, New York.

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Philip Odell, 108 Pleasant Street, Ayer, Massachusetts.

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Richard N. Williams, Apt. 7B, 15 E. Funstun Avenue, Spring Valley, New York.

William E. Wilson, 125 Fifth Avenue, Gloversville, New York.

ABSTRACTS

Chromatographic Fractionation of Rat Tail Tendon Collagen. A. Kessler, H. Rosen, and S. Levenson. *Nature*, **184**, 1640 (1959).—The portion of rat tail tendon collagen which is soluble in acetic acid at room temperature may be fractionated into 4 components by chromatography on carboxymethyl cellulose at 40°C. and elution with acetic acid. C.L.D.

Causes of Uneven Dyeing of Sheep and Lamb Fur Skins. J. Jansó. *Bőr és Cipőtech.*, **8**, 185–87 (1958); abst. in *J. Soc. Dyers Colourists*, **75**, 569 (1959).—With formaldehyde-tanned skins the main causes of uneven dyeing are too high an ironing temperature and uneven penetration of HCHO. In fat-liquoring, the main causes are uneven penetration and use of unsuitable greases. Mineral and vegetable oils and nonionic emulsifiers do not cause uneven dyeing, but sulfonated oils and soaps do with some colors. Urine stains that develop in light and pastel dyeings on panno-fix stains may be eliminated by treatment with H_2O_2 and/or hydrosulfite before dyeing. C.L.D.

Hydroxylysine in Wool. H. Kersten and L. Zürn. *Nature*, **184**, 1490–91 (1959).—The presence of hydroxylysine in wool was determined by paper chromatography, by paper electrophoresis, and by column chromatography. By the latter method the total hydroxylysine (sum of hydroxylysine and of allo-hydroxylysine) present in wool was found to be 0.20% by weight (0.218% of total N). C.L.D.

Effect of Ultrasound on the Protein of Human Serum Denatured with Tannin. W. Mizgalski and I. Zygmunt. *Nature*, **184**, 1436 (1959).—Human blood serum was treated with tannin and then examined by paper electrophoresis. Results indicated the tannin had formed an insoluble complex with the serum globulins, but not with the albumin. When the tannin-treated serum was subjected to ultrasound, part of the complex was broken up; the liberated α_1- and α_2-globulin fractions were increased slightly, while there was a slight decrease in the β- and γ-globulin fractions. C.L.D.

Model for the Helix-Random Coil Transformation in Polypeptides. L. Peller. *J. Phys. Chem.*, **63**, 1194–1206 (1959); abst. in *J. Soc. Dyers Colourists*, **75**, 576 (1959).—A partition function is used to describe configurations of a long polypeptide chain in aqueous solution. A thermal transition from a helical to a random coil configuration may occur. Increasing activity of a second component in the solvent interacting preferentially with random coil residues causes a transition to a random coil. The helix-random coil transition in a polyglutamic acid should result from increasing the pH and the transition is accompanied by considerable inoization of carboxyl groups. The possible significance of this type of ionization for anomalous titration behavior of some proteins is discussed. C.L.D.

Some Improvements in Colorimetric Determination of Elastin. V. Scarselli. *Nature*, **184**, 1563 (1959).—Experimental details are given of an improved method for the determination of elastin in tissue. The method is based on the photometric estimation of the amount of orcein liberated after the tissue, previously stained with the dye, is treated with elastase. C.L.D.

Optical Rotatory Power of Collagen. R. Burge and R. Hynes. *Nature*, **184**, 1562–63 (1959).—Soluble collagens, whose range of pyrrolidine content (proline and hydroxyproline residues) lay between 160 and 225 residues per 1000 total amino acid residues, were obtained by extraction with citrate-hydrochloric acid buffer (pH 3.7) of the swim bladders of cod and perch and of the skins of cod, herring, eel, and rat. The specific optical rotation for sodium D light is approximately −400° for each of the undenatured samples; it is therefore independent of the pyrrolidine content, and is fundamentally a property of the helix. For the thermally denatured samples in which the polypeptide chains are separated, each adopting a randomly coiled configuration, there is a systematic increase of specific optical rotation with pyrroline content. C.L.D.

Drying of a Mass of Hygroscopic Fibers by Forced Convection of Air: A Note on Some Physical Aspects. J. Downes and G. McMahon. *Textile Research J.*, **29**, 1006–10 (1959).—A theoretical discussion is presented which demonstrates that in drying a uniform mass of fibers by forced convection air flow through the mass, a "drying front" proceeds through the fibers in the same direction as, but much more slowly than, the air flow. For uniform drying, therefore, the process must continue until the front has emerged from the downstream face of the mass of fibers. Air of appropriate relative humidity must be used to leave the fibers at the desired regain. Reversal of air flow direction is undesirable on grounds of efficiency. C.L.D.

Dry Weight of Wool. I. Watt, R. Kennett, and J. James. *Textile Research J.*, **29**, 975–81 (1959).—The absolute dry weight of wool is defined as the weight obtained on heating the sample *in vacuo* to 140°C. for at least 10 minutes. Above 160°C. a dry wool sample loses weight because of degradation. From comparison with partially acetylated wool it is concluded that drying wool at low regains involved the removal of water from hydrophilic sites. C.L.D.

Animal-Glue Testing for Paper Sizing. P. B. Davidson and H. B. Bodenhagen. *Tappi*, **42**, 720ff (1959).—A standard laboratory tub sizing and air-drying procedure was developed that possessed reasonably good reliability and duplicated, as far as possible, the conditions that obtained in the mill processes. A numerical measure of penetration, based upon the determination of reflectance, was perfected which gave a statistical evaluation of sizing efficiency. Various testing methods were either adapted to or developed for

determining glue characteristics that might be pertinent to sizing efficiency. Some of these methods were found wanting and were discarded while others received further attention for the purpose of increasing their precision and reliability. A preliminary program was set up to test a large number of both hide and bone glues by the full list of test methods. From the results of this program nine representative hide glues were selected for more careful testing at both pH 7 and pH 9 by the refined methods. The results of these tests verified the supposition that gel strength and viscosity did not adequately define the properties of a glue for tub sizing of technical papers. The time required for formaldehyde to react with glue at a pH of 9 was very useful in differentiating bone from hide glues. The change in viscosity of a glue between pH 7 and pH 9 gave some indication to the class of glue which could be used for blending to give a specified gel strength and viscosity. The amount of water required to bring the viscosity of a 7.7% glue at 45°C. to the same viscosity at 25°C. was, in general, a good index of sizing efficiency. A sensitive surface-tension test, made with the Instron tester, not only produced a charted record of the surface tensions of the various glues but also gave a measure of the stretchability or toughness of the surface film on the glue. The full significance of this film elongation has probably not been realized, but this new measurement indicated the deleterious effects of zinc compounds, used as preservatives, upon glue. It is still uncertain whether the chromatographic method detects small concentrations of disintegration products in both hide and bone glues or just of bone glues. This much appeared evident, however: when such products were found to be present, the sizing efficiency of that particular glue was poor.

Authors' extract

Resin Tannages with Semitannins. G. Vágó, *Das Leder*, **10**, 272–76 (1959).—In resin tannages substances of small molecular size first penetrate the hide, then are condensed, but the resulting resins have little affinity for hide substance. If substances could be found that would condense to true tannins, they would change the skin to leather. Semitannins that might be used are mono- or divalent phenols, some of their monosulfonated products, catechols, and phenolcarboxylic acids. Semitannin, as used here, means compounds with only one active group; they can attach to hide but cannot form cross links. The compounds must be reactive enough to condense with aldehyde after some of their active groups have been lost by attachment to hide. Condensation may be made to occur in one of three ways: (1) with aldehydes, (2) with metal salts, or (3) by oxidative dehydration. The aldehyde condensation may be either the union of two or more molecules through methylene bonds or a Mannich condensation. Formaldehyde makes better leather than other aldehydes. For condensation with metal salts polycarboxylic acids are especially good. Salts of aluminum and zirconium are best; salts of divalent metals (Cu, Cd, Mn, Ni, Co, Zn, Hg) raise the shrinkage temperature, but the treated collagen has no decided leather character. Oxidative dehydration is the type of process used by plants. Various phenols were polymerized with peroxide in acetic acid and ammonium persulfate; only the beta-naphthol product caused leathering. During condensation with aldehydes there is unavoidably some aldehyde tannage. One cannot be sure, therefore, whether true tannins have been formed from the semitannins. To study this point hydration of the fibers was determined. A true tannage blocks some of the collagen groups that can hydrate.

The hydrophilic nature of the tanned fibers therefore should decrease even though the hydrophilic methylol groups of the methylolphenol increase affinity for water. Total water and bound water were determined both before and after thermal denaturation. Bound water was determined by drying at 105°C. samples from which all other water had been removed by high pressure. Samples were denatured by heating for three minutes at a temperature of 1°C. above the shrinkage temperature. Percentages of bound water before and after denaturation were as follows: unhaired skin, 64 and 97; formaldehyde-treated skin, 51 and 53; pyrocatechol-treated skin, 56 and 69; pyrocatechol-formaldehyde–treated skin, 52 and 50; and wattle-tanned skin, 51 and 35% resp. Although there was a decrease in bound water in the pyrocatechol-formaldehyde treatment, it was quite small in comparison with that caused by a vegetable tannin.

I.D.C.

The Effects of Soluble Salts on the Gelation of Tannin-Formaldehyde. K. F. Plomley. *Australian J. Appl. Sci.*, **10,** 294–97 (1959).—Gelation time, at pH 5 and 90°C., of a solution containing 33% of wattle or mangrove tannin and 3.3% HCHO, was reduced from 665 sec. to values ranging from 117 to 540 sec. by adding 3.3% of any one of a number of salts. Gelation time decreased with increasing salt concentration. In some cases the pH of maximum gelation time was shifted by adding salt, e.g., $CuSO_4$. The gluing properties of wattle tannin–HCHO were improved by adding 5% of Mn acetate, but not 10%.

H.B.M.

Studies on the Enzyme Depilation of Hides and Skins. III. The Influence of Fundamental Cultural Conditions on the Production of Protease and Depilatory Enzyme by Aerobic Bacteria. H. Toyoda and A. Futami. *Repts. Govt. Chem. Ind. Research Inst., Tokyo,* **54,** 369–77 (1959). **IV. The Influence of Constituents of Culture Medium on the Production of Protease by Bacillus megatherium 3E.** *Ibid.*, 378–90. (English summaries, *ibid*, pp. xLi, xLii; cf. abstracts, *JALCA*, **53,** 295, 533 [1958]; **54,** 54 [1959].)

H.B.M.

Shrinkage Phenomenon. V. Apparent Volume Shrinkage. N. Ramanathan, V. V. Subbalakshmi, and Y. Nayudamma. *Bull. Central Leather Research Inst., Madras,* **6,** 119–31 (1959).—Apparent volume shrinkage (AVS) was measured by determining apparent volumes with an Edwards densitometer (*J. Intern. Soc. Leather Trades Chemists,* **16,** 292 [1932]) equipped with a cathetometer for enhanced sensitivity. Specimens conditioned at 27°C. and 65% R.H. were measured for apparent volume, then soaked in water for 15 min., shrunk in water or 75% aqueous glycerol, dried, and reconditioned, and apparent volume was again determined. Area shrinkage (AS) (abstr., *JALCA*, **54,** 473 [1959]) was determined simultaneously on the same specimens. The shrunken and dried specimens were soaked in water for 24 hr., dried, conditioned, and measured a third time to determine volume recovery. Thickness of the dried, shrunken specimens was calculated from AVS and AS because

direct measurement was impossible owing to curling. The thickness may decrease, increase, or remain constant during shrinkage, depending on whether AVS is greater, less, or the same as AS. The leathers were strips of long-limed, delimed, acetone-dehydrated goatskin, tanned as in previous experiments (abstr., *JALCA*, **52**, 100 [1956]). Measurements on specimens of untanned pelt from many locations showed the mean AVS was 56% with a coefficient of variation of 3%, while mean AS was 34% with a coefficient of variation of 10%; thus AVS varies less with location than does AS. The values found for AVS and AS, in that order, as percentages of the original area or volume, for leathers variously tanned, were as follows: pelt (all locations), 56, 34; sulfonyl chloride, 47, 23; fish oil, 5, 9; Tannigan Extra A, 33, 37; basic Al sulfate, 36, 48; HCHO, 7, 7; wattle, 11, 39; Cr, 9, 8; Cr-wattle retan, 10, 20. AVS tends to decrease with increasing T_s and is less than AS for leathers containing much material deposited between the fibers, e.g., vegetable leathers. Pelt, and leathers tanned with Al, Cr, and Cr-wattle, showed no recovery of either volume or area; HCHO leather showed almost complete recovery of both AVS and AS; leathers tanned with Tannigan Extra A and wattle showed 41% and 49% recovery of AVS, but none in AS; leathers tanned with sulfuryl chloride and fish oil showed 30% and 47% recovery of AS but none in AVS. Speculations are advanced as to the significance of these differences. H.B.M.

Quantitative Determination of the Carbohydrate Constituents of Skin Proteins. K. Thomas Joseph and S. M. Bose. *Bull. Central Leather Research Inst.*, **6**, 132–43 (1959).—The proteins previously described (abstr., *JALCA*, **54**, 168 [1959]) were analyzed for total reducing sugars and for hexosamines. Determinations were made both on the hydrolyzate directly and after separating the sugars from peptides and amino acids by chromatographic techniques; the latter values were somewhat lower in the case of total reducing sugars, and very much lower in the case of hexosamines. Percentages found (after separation) for reducing sugars and hexosamines in that order were: collagen, 0.55, 0.31; citrate-soluble collagen, 0.12, 0.02; neutral salt–soluble collagen, nil, nil; reticulin (lymph node), 2.1, 0.15; reticulin (adipose tissue), 2.1, 0.13; elastin (*ligamentum nuchae*), 0.24, 0.07; albumin, 2.0, 0.45; globulin, 2.1, 0.56; mucoid, 6.4, 1.8. Galactose, mannose, glucose, and fucose were found in all the proteins; quantitative determinations showed that the first two were present in considerably larger amounts than the latter two. Glucosamine was identified in all the proteins, and elastin, albumin, globulin, and mucoid also contained galactosamine. H.B.M.

"Tanning" and "Detanning" Action of Dyes. G. Otto. *Rev. tech. ind. cuir*, **51**, 197–203 (1958); abstr., *J. Soc. Dyers Colourists*, **75**, 630 (1959).—Some leather dyes cause appreciable modification of the quality of the leather, but the effect in different tanneries may differ. It is not caused by over-neutralizing the leather. Some dyes have a tanning action as would be expected because their structure is similar to that of the syntans. Dyes having a tanning action and mol. wt. >350 improve the quality of the leather if the grain is not overtanned and if absorptivity of the leather is not increased so much that finishing becomes difficult. Instead of tanning, other dyes of lower mol. wt.

may form soluble compounds with Cr salts and partly detan leather. Dyes can enter the Cr complex, and their action may be modified by masking agents. Freshly precipitated $Cr(OH)_3$ was treated with four dyes of varying astringency and with masking agents. Formate, a weak masking agent, did not prevent takeup of dye or displace dye from dyed $Cr(OH)_3$. The strong masking agents, phthalate, oxalate, and hexametaphosphate, prevented takeup of dyes or displaced them. Citrate, adipate, malonate, and sulfophthalates were intermediate in action and displaced only the less astringent dyes. The undesirable effects of dyes on leather quality can be avoided by use, preferably during neutralization, of a masking salt with medium affinity for Cr. If the dye detans too much, it may be overcome by use of 60% basic $AlCl_3$, either during fixation of the dye or in the final stages of tanning. C.L.D.

Peptides Obtained from Partial Hydrolysis of Decalcified Human Dentin Collagen. C. C. Solomons. *Nature*, **185,** 101–2 (1960).—Decalcified human dentin collagen was partially hydrolyzed by heating with water in a sealed tube. Four peptides were obtained from the mixture by paper electrophoresis. One of these contained all the amino acids normally present in intact collagen; the second contained all these amino acids except hydroxyproline; the third contained all the amino acids except hydroxyproline, proline, and phenylalanine; the fourth contained only 7 amino acids. C.L.D

Enhancement of the High-spacing Meridional Reflexions in the X-ray Photograph of Keratin Impregnated with Heavy-Metal Salts. W. S. Simpson and H. J. Woods. *Nature*, **185,** 157 (1960). C.L.D.

Meridional Reflexions in the X-ray Diffraction Photographs of α-Keratin. W. J. Onions, H. F. Woods, and P. B. Woods. *Nature*, **185,** 157–58 (1960). C.L.D.

Effect of Age on the Heparin Content of Rat Skin. S. Schiller and A. Dorfman. *Nature*, **185,** 111–12 (1960).—The concentration of hyaluronic acid and of chondroitin-sulfuric acid in young rat skins is approximately twice that in mature animals. The heparin concentration diminished even more (75%). After 50 days of age, the level of all acid mucopolysaccharides in skin was fairly constant. C.L.D.

Wool Damage—Topochemical Evidence. K. Tomopulos and R. Ritter. *Melliand Textilber.*, **40,** 676–77 (1959); abstr., *J. Soc. Dyers Colourists*, **75,** 631 (1959).—The technique of the Pauly reaction applied to detect localized damage in wool (particularly alkali damage) is improved. Single fibers are treated first with LiBr solution of benzene-sulfonic acid diazonium salt and then with 5% to 10% aqueous Na_2CO_3; the damaged zones show colored coupling products. C.L.D.

Reversible Supercontraction of β-Keratose. F. G. Pautard and P. T. Speakman. *Nature*, **185**, 176–77 (1960).—Horse hair which has been oxidized with peracetic acid and extracted with NH_4OH will contract at lower pH and increase in length at higher pH; the changes are without hysteresis and can be repeated many times with a single fiber. The dimensional changes suggest at least a partial contraction to the cross-β configuration, although, since the X-ray diagram of the extended fiber is diffuse, not from a well-defined α-configuration but from some intermediate less regular state. C.L.D.

Bleaching of Fur Skins. M. Perlmutter. *Rev. tech. ind. cuir*, **51**, 1–6 (1959); abstr., *J. Soc. Dyers Colourists*, **75**, 625 (1959).—A discussion of the advantages of treating with HCHO to harden and improve the resistance to alkali of the hair. Surfactants give some protection to the hair during bleaching. C.L.D.

Variations in the Assessment of Lightfastness Exposures. K. McLaren. *J. Soc. Dyers Colourists*, **75**, 597–99 (1959).—Ten specimens exposed against the lightfastness standards by the ISO method were assessed by 72 observers, and the results were evaluated by statistical methods. The most important factor affecting the agreement among assessors was off-tone fading. The experience of the observer was generally quite unimportant. C.L.D.

Diffusion of Water into a Dry Keratin Fibre. M. Feughelman and M. S. Robinson. *J. Applied Polymer Sci.*, **1**, 371–72 (1959); abstr., *J. Soc. Dyers Colourists*, **75**, 622–23 (1959).—The stress was measured in a wool fiber held at fixed extension in water at 20°C. when it had reached a constant value (1 hr.), again after the water was replaced by dry Analar glycerol, and again after the glycerol was replaced by water. In the atmosphere of 0% relative humidity, the initial constant value increased rapidly and remained indefinitely at the high value. The original value was again found when the glycerol was replaced by water. The results are interpreted in terms of a two-phase picture of the wool fiber: a water-impenetrable phase and a matrix which is accessible to water. C.L.D.

Xenon-Arc Fading Lamps. K. McLaren. *J. Soc. Dyers Colourists*, **75**, 594–96 (1959).—Commercially available fading lamps based on xenon arcs were studied to determine how closely the nature of the light resembled daylight and the modification necessary to obtain the required effective humidity in fading tests. C.L.D.

Action of Nitric and Hydrochloric Acids on Wool with Particular Reference to Supercontraction of the Fibers. W. G. Crewther and L. M. Dowling. *Textile Research J.*, **30**, 23–36 (1960).—Treatment of wool with

40% HNO_3 at 0° to 30°C. for periods up to 18 hr. causes a decrease in sulfhydryl, disulfide, and tyrosine contents and an increase in free amino groups. Treatment with 28% HCl under similar conditions causes an increase in free amino groups, and under the more severe conditions, a decrease in tyrosine and an increase in disulfide. Alkali solubility and trypsin digestibility are greatly increased by either treatment. The kinetics of supercontraction are discussed in terms of chemical and configurational changes in the fiber. C.L.D.

Applicability of the Alkaline Thioglycollate Extraction Test for Detection of Wool Damage. K. Wegjan and H. G. Fröhlich. *Textile Research J.*, **30,** 73–4 (1960).—The alkaline thioglycollate extraction test (F. Lennox, *Textile Research J.*, **28,** 949 [1958]) is a highly sensitive test for the detection of acid damage to wool. C.L.D.

Dispersion of Wool Protein by Thiols in Acid Solution. W. E. Savige. *Textile Research J.* **30,** 1–10 (1960).—Wool is solubilized up to 50% by a 24-hr. treatment at 50° to 60°C. with a solution of thiol containing sufficient inorganic or organic acid to bring the pH to 2. The solubilized wool appears to be chemically unaltered except for reductive fission of disulfide bonds. Concentrated aqueous thioglycollic acid dissolves wool completely at 60°C. C.L.D.

Tests for Colour Vision. F. Jordinson and T. Minshall. *J. Soc. Dyers Colourists*, **75,** 585–93 (1959).—Various types of defective color vision and tests for their detection are discussed. The Wain test appears to be the best test devised for dyers. C.L.D.

Prevention of Mold Spots and Wet Surfaces in Leather Factories. K. Moritz. *Das Leder*, **10,** 276–78 (1959).—To prevent damp or moldy walls in rooms in which the relative humidity is high only a moisture barrier pasted to the plaster will be needed if insulation of the wall is adequate and there is no water-impervious layer on the outside of the wall to prevent drying out. If insulation is not sufficient, a fiberboard layer of some type will be needed between the wall and the moisture barrier. In this case the barrier is very important because at high humidities the fiberboard will absorb much water and lose its ability to prevent loss of heat. Vaporex is a 3-ply barrier material made in 50-cm.-wide rolls. It has a water-resistant center layer consisting of a gumlike film of isobutylene base and an outside (room side) layer of special paper that can take up or give off about 200 grams of water per square meter. I.D.C.

Progress in the Field of Enzymatic Unhairing. O. Grimm. *Das Leder*, **10,** 244–49 (1959).—A review. Fungal enzymes with activators have been used for many years for loosening the wool on sheepskins and also the hair

on goatskins and cattlehides. Only in special cases are carbohydrate-splitting enzymes used. Amylase, hyaluronidase, and pectinase will not loosen hair. During the past 30 to 40 years the enzyme-unhairing process has changed. Good loosening is now obtained in 12–24 hours at 20°C. instead of the 2 or 3 days at 30° or even 40°C. formerly required. Good leather can only be made by readjusting the soaking and bating steps to the unhairing step. An enzyme soak is best because it removes noncollagenous protein and thus increases rate of penetration of the unhairing enzyme. I.D.C.

Spruce Bark Tannin. XI. Polyhydroxystilbenes, a New Class of Condensable Tannins. W. Grassmann and H. Endres. *Das Leder*, **10**, 237–44 (1959); *see JALCA*, **54**, 302 (1959).—A lecture reviewing work on the structure of the principal components of spruce bark tannin. From the combined extracts of inner spruce bark with ethyl acetate and with alcohol was obtained 12% (based on total tannin in the inner bark) of diglucoside and 10% of monoglucoside of piceatannol, 3% of dihydropiceatannol-monoglucoside, 1% of trihydroxytetralin-monoglucoside, and 2% each of piceatannol and quercetin-glucoside. A water extract obtained after the ethyl acetate and alcohol extractions contained 50% of the total tannin, but no crystalline compounds could be obtained from it. Piceatannol was detected chromatographically in *Picea excelsa*, *P. glauca*, *P. amorica*, *P. orientalis*, and *P. mariana*. I.D.C.

Technological Problems of Vegetable Tanning. H. Herfeld. *Das Leder*, **10**, 285–93, discussion 293–94 (1959).—A lecture briefly reviewing standard processes for tanning sole leather in eight countries, and current investigations on rapid tannages. The important factors in rapid tannages are concentration of liquor and method of adding it, temperature, acidity, type of acid (mineral or organic), acid-salt ratio, type of pretannage or retannage, and use of a drum. A rapid 5-day tannage, based on the Russian process, made leather that was poor in color, density, strength, and wear resistance. I.D.C.

Interpretation of Investigations of the Tanning Velocity in Rapid Tannage of Heavy Leather. H. G. Wollenberg. *Das Leder*, **10**, 294–98, discussion 298 (1959).—A lecture. The time of tanning was reduced by a factor of 2.6 by raising the temperature from 8° to 38°C., and by a factor of 2 by increasing specific gravity of the tan liquor from 1.08 to 1.14. Important points for obtaining rapid tannage are as follows: (1) maintaining a rather large difference in concentration between the float and the interfibrillar spaces of the leather at all stages by frequent changes of liquor, (2) keeping the liquors in the various stages at as high temperature as permissable for the physical condition of the leather, (3) use of movement of liquor (the strongest liquor must be strengthened with a highly concentrated liquid extract, and the float-leather ratio should be as low as possible), (4) use of a drum at the intermediate stage, and (5) use of a suitable extract mixture of low viscosity. I.D.C.

Comparative Tests on Improving Loose Grain of "Rindbox" Leather. Ladislaus and Elizabeth Vermes. *Das Leder*, **10**, 267–71 (1959).—

Tests were made with 250-kg. packs of unhaired, split, frigorifico hides in order to determine the best process for producing leather with tight grain. Various modifications were made based on a standard chrome-tanning process. Comparisons were made of (1) a H_2SO_4-Na_2SO_4 pickle, (2) treatment with naphthalenesulfo acid instead of a pickle, (3) a HCl-NaCl-$Al_2(SO_4)_3$ pickle, and (4) pretanning with Tanigan CU instead of a pickle. The last two resulted in the tightest grain. The actual chrome-tanning process was modified by (1) adding 1.1% Implenal UR to the second and third (final) additions of chrome solution, (2) adding 2% polyethyleneglycol to the second chrome addition, and (3) adding 0.4 equivalent (chrome basis) of phthalic acid to the chrome liquor one week before use. No appreciable differences in grain tightness were noted. Comparisons were made of (1) pretanning with formaldehyde, (2) pretanning with polyphosphate, (3) a two-stage chrome tannage, and (4) a combination one- and two-bath process in which, after a sulfuric acid–alum pickle, the hide was treated with 10% chrome alum and 1.4% bichromate, then reduced with thiosulfate. In an additional check test the tightest grain was found after the formaldehyde pretannage. The alum pickle produced the next tightest grain and the best leather from all points of view. The combination one- and two-bath tannage produced strong, but loose, soft leather with a coarse grain. Retannage with wattle extract was not improved by addition of syntans. Optimum tanning conditions for obtaining a tight grain on Argentine hides are as follows: Lime with enough Na_2S to destroy the hair but with not more than 2% (hide basis, 400% float). Reduce milling to a minimum. For chrome tannage either use alum in the pickle, or pretan with formaldehyde. Retan with a combination of vegetable tannin (preferably wattle) and a syntan. Without syntan the grain is tender. Part of the oil should be given before and part after the retannage. I.D.C.

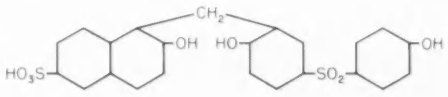
Data Regarding the Combination Chrome-Vegetable Tannage of Sole Leather under Laboratory and Factory Conditions. R. Ferebauer. *Das Leder*, **10**, 307–11, discussion 311–12 (1959).—Combination tannages of chrome with vegetable tannin or syntans are now used to speed the tannage of sole leather. Tannery tests of such retannages were made over a period of 20 months with daily 3000-kg. lots of salted medium-weight hides, and the results were published in Czechoslovakian journals. The most economical process was with from 2 to 4 suspenders and 2 phases. The leathers contained 0.8–1.0% Cr_2O_3, 27–28% organic matter (on fresh hide weight), and were satisfactory as regards analysis, color, abrasion, thickness, area, and use in the shoe factory. They had better water stability but were 10% weaker and had greater water absorption than normal leather, and the necks and flanks were distorted. Tannin loss in the spent liquor was 3–5% (fresh hide basis) as compared with a 2.3% loss by the normal process. Tests also were made to study leaching of chrome during retannage and other tanning factors. Four tan liquors were prepared by mixing a vegetable liquor with 0% (A), 30% (B), 45% (C), and 60% (D) of Syntan SP. The vegetable liquor contained 40% of sulfited spruce extract and 15% each of quebracho, wattle, chestnut, and oak. Syntan SP is a sulfonated novolak containing a peptizing, diphenolic resin. Tannages were made with each of these liquors on delimed hide and on hide pretanned with a 40% basic chrome alum (0.9% Cr_2O_3) liquor. During retannage there was a 12.7% loss of chrome (original chrome basis); the leather contained

0.06–0.93% Cr_2O_3. Weight yields decreased from 68.9% with A to 60.6% with D for unchromed leather, and from 63.6% to 60.8% for the retan leather; area yield, however, was the same for chromed and unchromed leathers. The retan leathers all had higher degrees of tannage, the same stitch tear strength, but lower tensile strengths than the unchromed leathers. However, as the proportion of syntan in the liquor increased this difference in tensile strength converged to zero. Increasing the proportion of syntan increased water penetration and decreased air permeability. No important differences between the unchromed and retan leathers were noted in the shoe factory. In a wear test, after a given time, 3 retan and 25 unchromed soles out of 51 of each required renewal. Laboratory abrasion tests were of no value in predicting wear life. Pretanning permits the use of more syntan in the blend. Leaching of chrome during retannage by 5 Czechoslovakian syntans was studied. Kortan Q1, a condensate of a naphthol-sulfonic acid with diphenols, and Syntan SP removed 10 or 15% of the total chrome used, while up to 30% was removed by some syntans under development which contained diphenols as their main component. Leaching increased as pH decreased. As each syntan increased in concentration leaching of chrome increased to a maximum, then decreased. I.D.C.

Modern Methods and Problems of Combination Chrome-Vegetable Tanning. S. G. Shuttleworth, R. L. Sykes, and D. Williams-Wynn. *Das Leder*, **10**, 299–306, discussion 306 (1959).—A lecture at the Munich conference reviewing the tannage and properties of chrome retan leather. Upper leather resistant to deterioration should be pretanned with a small amount of 33% basic chrome liquor; a masked chrome liquor, especially sulfophthalate, is good. Before retannage the leather should be neutralized or given a Calgon or syntan treatment. Retannage should be for a short time and with not too much vegetable tannin. Retannage of sole and insole leathers is also reviewed. I.D.C.

Isolation and Properties of Definite Constituents of Syntans. G. Reich. *Das Leder*, **10**, 261–66, discussion 266 (1959).—To determine the physical-chemical tanning laws substances of known constitution are required which are typical of the tannin group being studied and which can be prepared with the required purity in large amounts. A study was made of the condensation product formed from 2-naphthol-sulfonic acid, dihydroxydiphenylsulfone, and formaldehyde (it is identical with the former Gerbstoff QE of Bayer). The method of preparation was to sulfonate 1 mole of 2-naphthol with 1.5 moles of sulfuric acid by heating at 125°C. for one hour. The mixture containing the sulfonated naphthol was cooled to 100°C. and diluted with 200 cc. of water. After the addition of 0.8 mole of dihydroxydiphenylsulfone (D), 1.2 moles of formaldehyde was added over a period of two hours, and condensation was continued at 95°C. for an additional 6 hours. The sulfonated naphthol mixture was found by chromatography to contain 0.46 moles of free sulfuric acid, 0.875 moles of Schaeffer's acid (S) (2-naphthol-6-sulfonic acid), and 0.097 moles of the disulfonic acids, R acid (3.6) and G acid (6.8). The R_f values of the condensation products were as follows: R-R, 0.37, R-(G?), 0.48; S-S, 0.68–0.70; S-D, 0.84; and D.D-D, 1.00. To isolate the S-D product the sulfone (D) and its self-condensation product (D-D) were first removed

by thorough extraction with ether. The ether extract was mostly D and amounted to about 50% of the sulfone that was used. Then followed a stepwise precipitation of the aqueous solution with N-diethylaniline. Numerous tests showed that N-diethylaniline was preferable to alcohol, metal salts, or other amines for this purpose. A 0.25 molar solution of the diethylaniline in 2*N* acetic acid was added carefully to avoid an excess and with violent shaking. The precipitate was separated in a centrifuge and washed thoroughly with water. By repeated evaporation with ammonia excess diethylamine was removed, and the material was converted to the ammonium salt. By analysis, chromatography, and additional reactions the material was identified as 3-(2-hydroxy-6-sulfonylhydroxy-naphthyl-(1)-methyl-(1-4,4-dihydroxydiphenylsulfone.



From 129 grams of condensation product the following was obtained: 26 grams of ether soluble, 31 grams of amine precipitate, 12 grams of amine precipitate in the wash water, and 60 grams of nonprecipitatable matter. Tanning characteristics were investigated. Analysis by the filter method, metal salt precipitation, and amine precipitation gave purities of 100. Prolonged filter analyses were made also. In this new method the filtrate is collected in fractions up to a total volume of 500 or 1000 ml. Results are given as curves showing volume of filtrate (abscissa) plotted against percentage of dry matter in the filtrate (original solution basis). For tannins of high astringency the curves have low constant values; for tannins of low astringency the curves rise rapidly to a maximum, while the curves show a break if mixtures are present. The model substance (S-D) had a rather low astringency even though, from its constitution, is was not an auxiliary tannin. It was not as astringent as the original QE type condensation product from which it had been separated. Calfskin tanned with S-D had the following analytical values: combined tannin, 17.6%; degree of tannage, 31; shrink temperature, 74°C.; tanning value, 34; and combining value, 17. Corresponding values for calfskin tanned with QE were 28.5%, 59, 73°C., 65, and 33. Penetration by S-D was more rapid than by QE for 2 or 3 days, but it then stopped when the tannin was only 80% of the way through the hide. Even though sufficient tannin was present, it would not penetrate further. The rise in pH which accompanied tanning is suggested as the cause of this behavior. Practically, the results show that syntans with the best tanning properties are those which contain emulsified condensation products that are free from sulfonic acid groups and which contain only enough of compounds with sulfonic acid groups to maintain stability of the solution. I.D.C.

Mechanical Properties of Raw Collagen Fibers. F. Morgan and R. Mitton. *J. Soc. Leather Trades Chemists*, **44**, 2–23 (1960).—A systematic study is reported of the mechanical properties of acetone-dehydrated raw collagen fibers teased from cowhide. Fibers were mechanically conditioned and broken at known humidities, and the effect of varying the rate of application of the breaking load was studied. Breaking load and extension were determined with

an extensiometer. Because of the irregularity found in fiber cross sections, breaking length rather than tensile strength was calculated. This value was determined by dividing the weight per unit length of fiber into the load required to break the fiber. Mechanical conditioning did not alter fiber strength or elongation at break. Large differences in strength were observed when different test conditions were employed. Results obtained by varying the rate of loading from 3 g/min. to 24 g/min. suggest one mechanism of fiber failure at low rate of loading and another at the higher rate of loading. Fibers were strongest if equilibrated and tested in a humidity range of 40–70%. It does not appear possible to predict for any fiber the value of one property from a knowledge of other properties (i.e., strength from thickness or extensibility). J.M.C.

Zirconium Tannage. The Effect of Particle Size on the Penetration of Zirconium into the Pelt. D. Williams-Wynn and R. Sykes. *J. Soc. Leather Trades Chemists*, **44**, 23–32 (1960).—A diffusion technique was used to investigate the polymerization of zirconium chloride solution, the diffusion coefficient D being related by the proper mathematical equation to the radius of spherical particles. Particle size of Zr was studied as a function of time, concentration, and acidity. Particle size in solutions at the natural pH of the Zr salt increased rapidly with time, reaching a limiting value in approximately 4–5 weeks, and increased as molarity of the Zr salt decreased below 0.3*M*. Small additions of acid greatly reduce particle size, while alkali has the reverse effect. Pilot-plant experiments indicate that addition of dry Zr tanning salt to the drum, resulting in very concentrated tanning liquor, gives extremely rapid penetration. By this technique complete penetration of heavy hide was achieved with as little as 2% ZrO_2 on limed weight. J.M.C.

A New Waterproofness Tester for Gloving Leathers. R. Mitton and F. Morgan. *J. Soc. Leather Trades Chemists*, **44**, 32–41 (1960).—A machine similar to that of Mann (*JALCA*, **51**, 634 [1956]) is described. The rubbing action of Mann's machine is eliminated. Leather is pressed intermittently against a stationary wet surface. Tapping action is achieved by dropping a stainless ball through a height of 1 inch. Water penetration was detected electrically. Waterproofed glove leather required an average of 67 taps for penetration as compared with 10 for untreated leather. A correlation coefficient of $r = +.749$ was found when the laboratory test was compared with a practical trial in which gloves were used to pick up wet bricks. No significant correlation with other laboratory waterproofness tests was found, but the reproducibilty of the test was found to be very high. J.M.C.

Condensed Tannins. 3. Isolation and Estimation of (–) 7:3′:4′:5′ Tetrahydroxyflavan—3– 01, (+) Catechin and (+) Gallocatechin from Black Wattle Bark Extract. D. Roux and E. Maihs. *Biochem. J.*, **74**, 44–9 (1960).—Details of the isolation, characterization, and estimation of these catechins are given. J.M.C.

A Study of the Protein Impurities in Gelatins with Ion-Exchange Resins. A. Leach. *Biochem. J.*, **74,** 61–9 (1960).—The chromatographic behavior of several gelatins varied as to molecular weight (120,000–350,000) and isoelectric points (pH 5–9.4) was examined. The proportion of gelatin eluted from the column is shown to depend on column loading, pH and ionic strength of eluting buffer, surface area of resin, and type and molecular weight of gelatin. A small proportion (0.36%) of a protein-polysaccharide complex (suspected to be derived from mucoprotein) was isolated from lime-processed gelatin. The view that lime-processed gelatins contain 10–19% of a second, very different, component is not confirmed. A modification of the Neuman-Logan hydroxyproline test is used in which oxidation is carried out at 40°C. to insure destruction of excess H_2O_2. J.M.C.

A Method for Assessing the Relative Cutting Properties and Abrading Properties of Leathers. N. Ramanathan. *Bull. Central Leather Research Inst., Madras,* **6,** 169–72 (1960).—Specimens 2" x 2" were mounted to form a continuous band on the periphery of a revolving drum, which was brought in contact with the knife blade or a piece of abrasive paper mounted on an arm. Thus all specimens were exposed to the same treatment. Ease of cutting was estimated from the depth of the knife cut. Relative ease of cutting and resistance to abrasion are given for leathers tanned with different tannins. Apparently these two properties are not related. H.B.M.

Studies of Enzymes for Leather Manufacture. IV. The Keeping Quality of Enzyme Bates, Depilants, and Other Enzyme Preparations. S. C. Dhar and S. M. Bose. *Bull Central Leather Research Inst., Madras,* **6,** 173–82 (1959).—The materials were stored dry at 4°, 25°, or 37°C., and proteolytic activity was determined at intervals of 90 days, or 30 days for purified materials, using Anson's method (see abstract, *JALCA*, **50,** 642 [1957]). Depilant M (a mixture of dried and ground *A. parasiticus*, together with the medium on which it was grown, and ground pancreas) lost 8% of its activity in 180 days at 4°C., 5% in 90 days at 25°C., and 8% in 90 days at 37°C. Losses after 2 yrs. were 36, 46, and 51% respectively. Enzyme bates No. 1 and No. 2 (the above enzyme mixture plus $(NH_4)_2SO_4$ or NH_4Cl) were considerably more stable than Depilant M, particularly at the lower temperatures. Tests showed that the stability of the ground mold powder was enhanced by mixing it with $(NH_4)_2SO_4$. When the enzyme was extracted from the mold powder and precipitated, its stability was enhanced by mixing it with wood flour, but dialyzing the precipitated enzyme resulted in loss of stability. Enzyme products obtained by precipitation with acetone or alcohol were considerably more stable than the purified enzyme previously studied. H.B.M.

Investigations on Pelt Swelling. I. Kerese. *Bőr- és Cipőtechnika,* **9,** No. 1, pp. 11–15, (1959); *Hung. Tech. Abstr.*, **11,** 136 (1959).—A new method for measuring the tautness of pelts and some of the fundamental questions of lime swelling are discussed. Pelt tautness is best characterized by compressive forces from 0.5 to 5 kg. per sq. cm. A linear relationship exists between the logarithms of compressive forces and the deformations promoting them. The

degree to which the pelts are limed is not characterized adequately by the water content alone; better results are obtained by measuring the tautness. The quality of the leather is greatly affected by the tautness of the pelts.

Author's abstract

Experiments on the Loss of Weight of Hides in the Presence of Sodium Carbonate. A. Simoncini, *Cuoio, Pelli, Mat. concianti*, **34**, 424–38 (1958).—Among the methods of denaturation of NaCl used for salting hides and skins, that with Na_2CO_3 and naphthalene is the most common in Italy. Some experiments were made to determine if this method causes loss of weight in green hides. The amount of Na_2CO_3 added to several lots of hides was 0 to 5%; the amount of naphthalene was always 1%; the hides were salted with 35% salt, piled in rooms at ordinary temperature and at 80% relative humidity, and examined after 2 and after 4 weeks. Hides salted with 2–3% Na_2CO_3 in the salt showed losses of 2.5–3.5%; those salted with greater amounts of Na_2CO_3 in the salt showed losses of 5% maximum. Only 8% of the hides had little red or violet stains. Analyses of the hides gave a progressive decrease of the Me_2CO extract with increased Na_2CO_3, and this fact is probably due to the increasing saponification of the fatty matter contained in the hides.

G.A.B.

Contribution to the Knowledge of the Constitution of Sumac Tannin. I. E. Durio, A. Catino, and A. Oddone (Univ. Turin, Italy). *Cuoio, Pelli, Mat. concianti*, **35**, 183–94 (1959).—Sumac extract (from leaves of *Rhus coriaria* of Sicily) was successively extracted with $CHCl_3$ (to eliminate chlorophyll), Et_2O (to eliminate flavones and related compounds), and Me_2CO. The acetonic extract was purified with Et acetate, MeOH, and EtOH, and fractionated by chromatography. Some fractions were obtained giving the reactions of pyrogallol tannins, but exact identification was not possible because the substances were not sufficiently pure. G.A.B.

Thickness of Leather and its Measurement. G. D'Orsi and M. Jannelli, *Cuoio, Pelli, Mat. concianti*, **35**, 9–30 (1959).—Experiments made with special types of variable-pressure gauges show that at a pressure of 2150 g. per sq. cm. sole leather has a maximum change in thickness of 0.8%; chrome leather, 5–6%; vegetable insole leather, 3.3%; upper leather, 4.7%; and chamois leathers, up to 43%. For the exact measurement of thickness and to obtain uniform results, it is proposed to have a gauge with (1) jaws no longer than 500 mm.; (2) circular, plane jaw feet, 1 cm. in diameter; (3) a dial plate divided into 0.01 mm.; and (4) a pressure spring giving a constant pressure of 500 g. per sq. cm. at any thickness of the leather. A special device is described to maintain this constant pressure of the gauge. G.A.B.

Chlorine Tannage. A. Simoncini, *Cuoio, Pelli, Mat. concianti*, **35**, 167–81 and 269–83 (1959).—Because some halogen derivatives insolubilize gelatin, the behavior of HClO, NaClO, $NaClO_2$, and chlorine water on hides was

examined. These products insolubilize gelatin in a nonuniform way, probably because there is a rapid insolubilization of a surface layer, which protects the underlying layers. Tanning experiments with Cl water (at a concentration of 4 g. active Cl per l.) in the presence of NaCl gave white soft leathers with T_s about 75°, but these leathers are detannized by washing with a solution of Na bisulfite. With hypochlorous acid (5 g. active Cl per l.) pale yellow, soft leathers were obtained, with brilliant grain. Washing with $NaHSO_3$ reduces the content of combined Cl but does not detan the leathers completely. Experiments with NaClO gave leathers of a good color and fullness both at pH 9 (when only the salt is present in the solution) and 5.6 (when the hypochlorous acid predominates). $NaClO_2$ has only a poor tanning action. Chlorine tannage made with buffered Na hypochlorite solutions is suggested for pretanning hides to be later tanned with chrome or vegetable tannins. G.A.B.

Practical Notes on Tanning Pickling Leathers for the Textile Industry. G. A. Bravo. *Cuoio, Pelli, Mat. concianti*, **35**, 31–36 (1959).—To obtain leathers with high flexibility and tensile strength and low elongation, it is necessary to choose hides without defects, perfectly unhaired, and unsplit (in order not to decrease the resistance of the fibers). Complete deliming (with NH_4Cl or lactic acid) and a bate which is not too strong are useful. Tanning starts with vegetable baths at 0.5° Bé. (1 day), increasing to 2°–2.5° Bé in a week; after 2 weeks, the hides are placed in drums at 8° Bé. (1 day) and 10° Bé. until they are completely tanned (1–2 days). The degree of tannage, even at complete tannage, is not so high as for sole leather. The extract mixture is commonly made with sweet (or sweetened) tannins. Fatliquoring is done so that the hides retain a minimum of 20% fatty matter; generally the mixture is made with 1/3 paraffin, 1/3 stearin, and 1/3 degras. Drying is very slow. If the tannage is regular, the hides show a minimum tensile strength of 3.5 kg. per sq. mm. G.A.B.

Provisional Methods of Analysis Used in the Italian Leather Experimental Station of Naples. *Cuoio, Pelli, Mat. concianti*, **35**, 195–209; 289–318; 424–57; 504–50 (1959).—The following methods are described with details: analysis of vegetable tannins; chemical and technological detection of tannins; precipitation reactions (with HCHO, Br water, Pb acetate, methylurethane, $(NH_4)_2S$, Fe^{+++} salts, Co salts, $KCrO_4$, concd. H_2SO_4, Schell Reaction, etc.); indexes for characterizing tannins (AcOEt solubility, EtOH solubility, Mo index, furfural index); fluorescence examination; cellulose sulfite detection (Procter-Hirst Method, Appelius-Schmidt Reaction); detection of synthetic tannins; detection of sulfated oils in tannin extracts; detection and determination of SO_2; tannin determination by various methods (Lowenthal, Wilson-Kern, Official Shake Method, Filter-Bell Method); sampling and preparation of solutions for analysis; extraction of vegetable matters; determination of H_2O, total solid matter, soluble matter, nontannins; semimicro method of nontannin determination; sp. gravity; reducing sugars; pH; sediments; ashes (with determination of Cu and Fe); tanning and binding power; tanning assays. G.A.B.

The Action of the Natural Acidity of Chestnut Extract in Comparison with That of Artificially Acidified Extracts. C. Bellucci-Sessa (Soc. Ledoga, Milan, Italy). *Cuoio, Pelli, Mat. concianti*, **35**, 420–23 (1959).—Extracts, e.g., chestnut, which contain a certain amount of organic acid behave during tannage in a very different way from sweet cathechuic extracts. Absorption of tannin by hides is due to many factors, among them astringency, which depends even on the natural acid character (due to the –COOH groups) of the tannin molecule. Artificial acidification of sweet extracts does not introduce –COOH groups in the molecule, while these groups are naturally contained in the chestnut extract and may even be neutralized (in the preparation of sweetened extracts) without changing the structure of the molecule.

G.A.B.

Research on the Constitution of Eucalyptus Wood. L. Capitó and L. Panizza. *Ann. chim.*, **49**, 271–81 (1959).—Among plants cultivated in Italy for paper pulp, *Eucalyptus camaldulensis* or *rostrata* is very well acclimatized. Some research is now being conducted on the constituents of various parts of the plant. The $(CH_3)_2CO$ extract of powdered wood heart (6.5–7.5% on dry basis) was fractionated by successive extractions with petroleum ether (1–1.5%), ethyl ether (15–20%), and ethyl acetate (av. 8%). From the ethyl ether extract, crystallized *d*-cathechin was obtained, and from the crystallization waters, ellagic, protocathechuic, and gallic acids were recovered. From the ethyl acetate extract a substance was obtained which by alkaline melting gives phloroglucin and protocathechuic acid, and by heating with HCl gives cyanidin chloride; therefore, it is supposed that this substance has a structure like that of leucocyanidin. By chromatography it was even possible to identify delfinidin chloride, which gives phloroglucin and gallic acid by scission. G.A.B.

The Importance of Quality Control in the Various Phases of Sole Leather Manufacture. N. De Pisapia (Soc. Ledoga, Milan, Italy). *Cuoio, Pelli, Mat. concianti*, **35**, 407–19 (1959).—Quality control in sole leather manufacture is necessary to improve quality, in order to win the competition with leather substitutes. G.A.B.

Hides and Leather in Ancient Greece. G. A. Bravo. *Cuoio, Pelli, Mat. concianti*, **35**, 89–111 (1959).—Some items on tanning and leather in Cretan and Minoan times are reported. Quotations referring to leather in the Iliad, Odyssey, and other Greek works are recorded, together with historical data on the most celebrated Grecian tanners and shoemakers. G.A.B.

The Causes of Yellowing of White Leathers. R. Stratta and E. Meda. *Conceria*, **67**, No. 2800, 14–17 (1959).—White leathers sometimes yellow not only because of the action of light, the diffusion of colored compounds from the inner layers to the surface, impurities contained in the glue, plasticizers, synthetic tannins, etc., but also because white pigments (TiO_2, ZnO, lithopone, etc.) are found to contain traces of metals, e.g., Fe, 0.01–0.012%; Mn, 0.03–0.04%; Pb, 0.012–0.025%; and Cu, 0.01–0.013%. G.A.B.

The Nontannins of Tanning Extracts. G. A. Bravo (Soc. Ledoga, Milan, Italy). *Cuoio, Pelli, Mat. concianti*, **35**, 395–405 (1959).—The constituents in tanning extracts and baths are reviewed, and their action during tannage is discussed. Nontannins are generally necessary to regulate the absorption of tannin by hides and its combination, particularly in surface layers, but they should not be present in too high amounts. Control (analysis, buffer power, pH, salt-acid ratio, etc.) of tanning baths is always necessary in order to be able to correct in good time unexpected changes in conditions of the tannage which may affect the uniformity of quality of the leather. G.A.B.

Retannage of Vegetable and Chrome Leather with Methylol Melamine. Y. Hachihama and Y. Kyugoku. *Technol. Repts. Osaka Univ.*, **9**, Nos. 337–62, 221–27 (1959) (In English).—Delimed calfskin was tanned with a mixture of vegetable tannins, or with a commercial Cr tanning material. The leathers were retanned with methylol melamine (MM) (about the pentamethylol derivative) or with the methyl ether (MeMM) (about 50% etherified). Retannage was carried out at pH 4 for 3 days, then at pH 6 for 3 days, and then the leathers were neutralized. In the case of vegetable leather, retannage with MM and MeMM raised the shrinkage temperature from about 83°C. to 104–115°C. and 96–105°C., respectively; decreased H_2O-soluble matter from 17% to 2% and 3%; increased sp. gr. from 1.37 to 1.50 and 1.49; and decreased elongation at break and tensile strength by about 20%. Retannage with HCHO had a considerably smaller effect on shrinkage temperature and H_2O-soluble matter, and very little effect on sp. gr. In the case of Cr leather, retannage with MM and MeMM increased shrinkage temperature from 81°–84°C. to 94°–98°C.; increased sp. gr. very slightly; and reduced elongation at break by as much as 50% in some cases, while tensile strength sometimes was unchanged but sometimes reduced by 50% or more. Water absorption of both leathers was reduced by varying amounts (up to about 20%) by retannage with MM and MeMM, but not by retannage with HCHO. It is thought that the increased shrink temperature is due to combination of MM or MeMM with collagen, the increased density to deposition of condensed MM or MeMM, and the decreased H_2O-soluble matter to reaction with unfixed tannin.
H.B.M.

Determination of Hydroxyl Groups in Some Raw Materials for Syntan Production. F. Langmaier and P. Symersky. *Kozarstvi*, **9**, 241–44 (1959).—In Czechoslovakia the best materials for syntan production are technical mixtures of biphenols, e.g., Optol and Difen. The Ogg-Porter-Willits modification of the Verley-Bölsing method was used. The sample of biphenol or syntan is weighed into a bottle with 5 ml. of a 1:3 mixture (by volume) of acetic anhydride and pyridine, the glass stoppers are moistened with pyridine, the well-stoppered bottles are heated 45 min. on a boiling water bath, 10 ml. water is added, and the mixture is heated 2 min. more to decompose excess acetic anhydride. After cooling, the stopper and walls of the bottle are washed down with 10 ml. butanol, and the solution is titrated with alcoholic NaOH. A blank is run simultaneously. Pure phenols, such as phenol or pyrocatechol, can be titrated directly, using neutralized cresol red indicator, but technical

phenols and syntans must be titrated potentiometrically because of the dark color. OH groups are calculated on the C content of the material. The Aulin-Erdtmann spectroscopic method and the Goldschmid differential spectrographic method are discussed. They are more rapid than the acetyl method but require a judicious choice of standards. Determinations of OH in Difen, Optol, and two syntans by the acetyl and differential spectrographic methods were in fair agreement except in one syntan, where the spectrographic method gave a value that was 50% too high, because of unsuitable choice of standard. L.M.

Histology of Corium of Horsehide Shell. A. Frank. *Kozarstvi*, **9**, 232–36 (1959).—The shell layer of horsehide butt has been studied microscopically. A new form of collagen fibers, described as "spruce structure," is reported. A film has been found at the boundary of the reticular and papillary layers, composed of spruce structure fibers and elastin. The "summits" of the spruce structure fibers touch this film, and their "branches" fill the shell layer. The boundary of the shell toward the flesh side is formed by parallel layers of spruce structure fibers. L.M.

The Chromatography of Phenolic Substances. J. Strachota. *Kozarstvi*, **9**, 276–78 (1959).—The column chromatography of phenolic substances is described. The elution capacity of O-containing adsorbents depends directly on the polarity of the solvents as deduced from their dipole moments or dielectric constants. 153 references. L.M.

Hygienic Problems with Technical Acetone and Acetone-Containing Solvents. M. Zeman and M. Klatil. *Kozarstvi*, **9**, 279–80 (1959).—Methods of analysis of acetone and solvent mixtures containing it are described. The polarographic method for acetaldehyde gave positive results with all types of acetone; even so-called C.P. acetone gave positive Shiff and Tollens reactions for acetaldehyde. Technical acetone and solvent mixtures containing it contained 1.8–2.1% aldehyde as acetaldehyde. Some of these mixtures are hygienically tolerable; some are not. The most dangerous solvents are methanol and methyl acetate. A mixture of 40% acetone, 30% methanol, and 30% methyl acetate, containing as little acetaldehyde as possible, is considered unobjectionable, assuming good ventilation. L.M.

Process Control of Vegetable Tannage. L. Masner. *Kozarstvi*, **9**, 181–84 (1959).—A review with 33 references. L.M.

Technological Possibilities of Enhancing the Effectiveness of Tanning Sole Leather. K. Müller and Z. Kotasek. *Kozarstvi*, **9**, 195–97 (1959).—Solvent tannage is discussed. Water, present in the gel of raw hide, impedes the passage of tannins and air. Quicker tannage is possible by removing water.

but ordinary drying causes coalescence of fiber bundles, so water must be removed by solvents (acetone or lower alcohols) or by freeze-drying. Chen (*Leather Mfr.*, **63**, No. 6, 3–19 [1952]) proposed pretanning of pelt by a naphthalene syntan, followed by heating in vacuum to 50°C. and sucking through a solution of mangrove extract at 50°C. Tannage was complete in 5 min. The quality of sole leather depends also on tension during tanning. Fixation of tannins depends more on the type of solvent than on the kind of tannin. Pelt fixes more acids and more Cr salts from acetone solution than from water, but the absorption of Cr is reversible. Dehydrated pelt fixes more HCHO (gas) than it does from aqueous HCHO, but less than from acetone solution. Solvent tannage is complicated but permits the use of H_2O-insoluble condensates of catechol tannins and HCHO, which are simpler to manufacture than analogous water-soluble condensates (commercial syntans). L.M.

Novelties in the Technology of Vegetable Tanning. L. Masner. *Kozarstvi*, **9**, 198–202 (1959).—A review. Hot pit tannage is recommended also for Cr-vegetable tannage, but temperatures of around 50°–55°C. are needed. 43 references. L.M.

Determination of the "True" Reducing Power of Some Substances Used in the Reduction of Chrome Compounds. L. Del Pezzo (Staz. Sper. Ind. Pelli, Naples, Italy). *Cuoio, Pelli, Mat. concianti*, **35**, 380–94 (1959).—The reduction of $K_2Cr_2O_7$ with glucose for the preparation of chrome-tanning baths is not a stoichiometrical reaction, because the theoretically calculated amounts of glucose are not sufficient to get the complete reduction. Following the reaction scheme given by Stiasny, 1 mol. $C_6H_{12}O_6$ is reduced by 4 mol. of $K_2Cr_2O_7$; but in practical experiments an amount which is 148.65% of the theoretically calculated amount is necessary. An experimental curve was recorded which makes it possible to find the quantities of pure glucose necessary to reduce $K_2Cr_2O_7$ completely, and, under the same standard conditions, even to calculate the reducing power of any other reducing substance (calculated as glucose). This method, giving results noticeably different from those given by the Fehling method but easier in its execution, is the following: 5 cc. of an approximately 0.1% solution of the reducing substance, with 10 cc. of 0.1*N* $K_2Cr_2O_7$, and 50 cc. of 6*N* H_2SO_4 is boiled with reflux condenser for 2 hr.; a blank test is prepared and boiled for the same time; after cooling, the solutions are titrated with 0.02*N* $Na_2S_2O_3$; the amount of $K_2Cr_2O_7$ reduced is calculated; and with the aid of the standard graph, the exact reducing value of the substance is known. The curve is drawn as the graphical record of the mathematical expression

$$y = 15.40\left(1 - e^{-\frac{10.08}{x}}\right)$$

where y = cc. of 0.1*N* $K_2Cr_2O_7$ and x = mg. (pure) glucose. G.A.B.

The Advantages of Purification and Filtration of Air in Tanneries. *Conceria* No. 2790, 33–36 (1959).—It was observed that the fine dust produced during tannery operations, when spread into the air, may produce damage to neighboring gardens, houses, and other factories. The dust contained in the air is particularly injurious to tanneries drying varnished or enamelled leathers in the open air. Tannery machines producing the greatest amounts of dust are the brushing and buffing machines: a brushing machine displaces 80–90 cu. m. air per min., and a buffing machine of 1500 mm. displaces 160–180 cu. m. air per min. It is necessary to eliminate the dust without getting new air from outside, which is too cold in winter. This is accomplished by using air filters, which send purified air back into the same room from which it is drawn. The main difficulty is the choice of filter: Those of jute, cotton, or carpet-type wools are easily obstructed, and the pressure necessary for circulating the air increases from 40–50 to 200–250 mm. Fabrics made with Perlon are suitable, economical, and may work either under pressure or vacuum: these fabrics should have a coarse or fine texture, according to the type of leather to be prepared. Crust leather buffed with a coarse-grained emery cloth produces a coarse dust, and the filters may have a more open texture, while for dust produced in the manufacture of chamois leather, filters with less open texture are used. G.A.B.

Tannins as Denicotinizing Agents. A. Simoncini, *Cuoio, Pelli, Mat. conciant.*, **35**, 83–88 (1959).—The reaction of precipitation of alkaloids is used to eliminate nicotine from tobacco smoke in filters for cigarettes: the filters are prepared with powdered wood or leaves of plants containing tannin, or with powdered tannin extracts, alone or in mixture with silica gel, active carbon, cellulose, etc. Chemical examination of the gases at the end of the filters shows a decrease of 60–65% in nicotine and all tar matters contained in the smoke. This invention was patented with Italian Patent No. 588,105 of Jan. 31, 1959. G.A.B.

Notes on a New Auxiliary Product for Leather Manufacture. R. Allegrini (Soc. Ledoga, Milan, Italy), *Cuoio, Pelli, Mat. conciant.*, **35**, 497–503 (1959).—Compounds of the general composition COOMe · R′ · COOR″ (where R′ = aliphatic, aromatic, or heterocyclic radical; R″ = alkyl radical with C_7 to C_{20}, both satd. or unsatd.; and Me = H, alk. metal, or organic basis) have a good bleaching, filling, and softening action on chrome or vegetable leathers. In chrome tanning, these compounds may be added to the chrome liquor or to the neutralization bath; in vegetable tanning or retanning, they may be added to the tannin mixture in the drum. The chemical and physical analyses of leather treated with these products are not changed. G.A.B.

Curing Hides and Skins with Salt Containing Antibiotic Substances. Z. Bayer, E. Ludviger, and V. Johanides, *Cuoio, Pelli, Mat. conciant.*, **35**, 477–85 (1959).—Several antibiotic products were tested during experimental curing of hides and skins (and even wool) with salt, i.e., penicillin, chlorotetra-

cyclin, oxytetracyclin, and chloramphenicol. The latter gives particularly interesting results, because the addition of 15–30 mg. of product to 1 kg. salt permits a decrease in the amount of salt to 15% on the raw hide weight without damage to the cured hides, while the physical properties of box calf prepared from these hides were better than the normal ones. G.A.B.

Relation Between Configuration of Fibrous Proteins and Their Chemical Structure. N. S. Andreeva. *J. Polymer Sci.*, **38,** 565 (1959); abstr. in *J. Soc. Dyers Colourists*, **76,** 54 (1960).—Departure from a completely random arrangement of amino acid residues in the protein chain gives rise to specific configuration in individual chain fragments. C.L.D.

New Artificial Weathering Methods. G. F. Bush. *Am. Dyestuff Reptr.*, **49,** 33–39 (1960).—The following factors in artificial weathering are considered: sunlight, moisture (including rain), temperature, gases, nuclear radiation, dust, and wind. Instrumentation for controlling some of these quantities at preset values in the test space is discussed. C.L.D.

Nature and Cause of "Canary" Coloration of Wool. J. A. Serra and R. M. Albuquerque. *Publ. fac. cienc. univ. Lisboa, Sec. A., Cient. invest.*, No. 5 (1956); abstr. in *J. Soc. Dyers Colourists*, **76,** 54 (1960).—Yellow discoloration of wool is caused by the action of alkaline suint on keratin, forming β-thiobis-(α-aminoacrylic acid). C.L.D.

An Elastin Gel. N. H. Grant and K. C. Robbins. *Nature*, **185,** 320 (1959).—Elastin (obtained from cattle aorta by treatment with 1*N* NaOH to remove collagen) is treated with elastase and then suspended in 0.2*N* acetic acid; the suspension forms a gel on standing. C.L.D.

Oxidative Dyeing of Wool. A. I. Levchenko, *Tekstil. prom.*, **19,** 48 (1959); abstr. in *J. Soc. Dyers Colourists*, **76,** 57 (1960).—Furs, wool, and unions can be dyed with amino derivatives of carbazole, dihydroxynaphthalenes, and aminocarboxylic, hydroxycarboxylic, and aminohydroxysulfonic acids, using $K_2Cr_2O_7$, $CuCl_2$, $NaClO_3$, or H_2O_2 to form a quinone dye on the fiber. C.L.D.

Esterlike Groups in Different Collagens and Gelatins. J. Bello. *Nature*, **185,** 241 (1960).—Esterlike groups in commercial acid-precursor pigskin gelatin and in bovine hide corium collagen have been found by reaction with hydroxylamine. C.L.D.

Amino Acid Analysis of Cuticle-Rich Material from Wool. J. Bradbury. *Textile Research J.*, **30,** 128–35 (1960).—Cuticle material of various degrees of purity was obtained from three samples of wool by descaling. The

material consisting of about 50% cuticle contained on the average 27% more proline and 40% less aspartic plus glutamic acids than the parent fibers for each of the three samples. In one sample the cuticle-rich material was richer in glycine (39%), and in another richer in cystine (22%), than the parent fibers.
C.L.D.

Supercontraction of Keratin Fibers by Lithium Bromide. A. E. Brown and L. G. Beauregard. *Sulfur in Proteins, Proc. Symposium, Falmouth, Mass.*, **1958**, (1959) 59–71; abstr. in *J. Soc. Dyers Colourists*, **76**, 130 (1960).—Supercontraction of human hair in aqueous LiBr does not involve disulfide bonds and can be explained in terms of secondary bond breakdown. Cleavage of H bonds between peptide groups, and of secondary bonds involving COOH and NH_2 groups, occurs.
C.L.D.

Some Multivariate Statistical Techniques Used in Color Matching Data. J. E. Jackson. *J. Optical Soc. America*, **49**, 585–92 (1959); abstr. in *J. Soc. Dyers Colourists*, **76**, 140 (1960).
C.L.D.

Influence of Shadow Quality on Color Appearance. S. M. Newshall, R. W. Burnham, and R. M. Evans. *J. Optical Soc. America*, **49**, 909–17 (1959); abstr. in *J. Soc. Dyers Colourists*, **76**, 140 (1960).—Shadows of skylight quality and of daylight quality were cast upon various samples. The color of each sample, both shadowed and unshadowed, was matched by adjusting the field of a visual colorimeter. Only skylight shadow evoked any trend in hue, and this was toward blue; but both shadows produced systematic losses in saturation and lightness.
C.L.D.

Calculation of Small Color Differences. F. L. Wurzburg, A. DiBermardo, and J. R. Cleary. *J. Optical Soc. America*, **49**, 974–80 (1959); abstr. in *J. Soc. Dyers Colourists*, **76**, 140 (1960).—Photoelectric colorimeters used as differential instruments determine the ratios of tristimulus values of similar colorants to a high degree of precision. Lengthy methods of calculating color coordinate differences from such data are avoided by the use of an approximation.
C.L.D.

Fluorescence and Gray Content of Surface Colors. R. M. Evans. *J. Optical Soc. America*, **49**, 1049–59 (1959); abstr. in *J. Soc. Dyers Colourists*, **76**, 140 (1960).
C.L.D.

ERRATA

In the May issue, p. 307, 4 lines from the end of the abstract on "Theory and Practice etc.", the temperature should read "270°–290°" *vice* "270°–270°". On p. 309, line 6, "Propen OR" should read "Oropon OR".

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New Esterified Oils Offer Major Benefits To White Tanners

An important development was announced recently by a leading manufacturer of tanning chemicals.

After considerable basic research, Seaboard Chemicals, Inc. is the first to offer the tanner a new concept in white leather fatliquors. It's a series of esterified oils called the Polarsols.

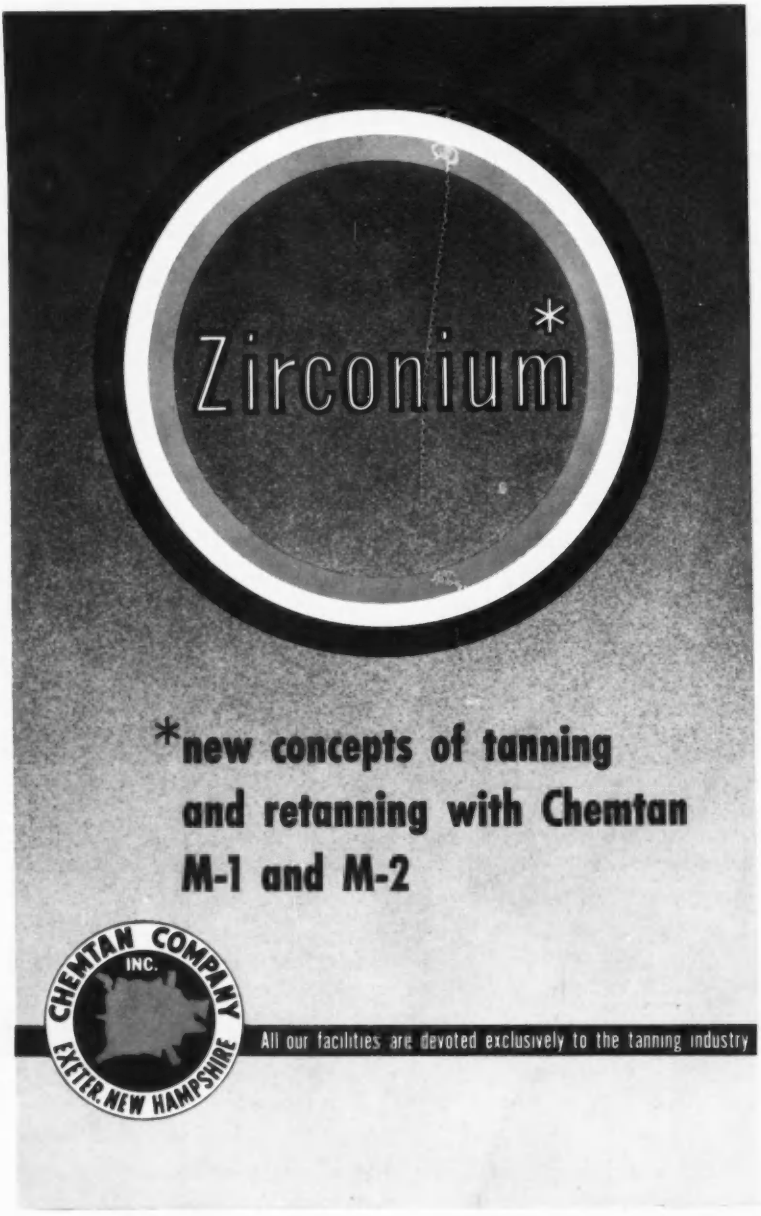
Tannery production runs indicate that these oils possess an attractive combination of properties that can help white tanners get more top-grade leather per pack and those already using the Polarsols claim the temper of their stock can be kept under absolute control, from mellow all the way to super-soft.

Unlike ordinary sulfated oils, which tend to produce looser leather as oil percentage increases, Seaboard's new esterified oils produce an overall tight break regardless of oil percentage used. Reports from the field indicate this is true even out into the flank and belly areas, with the result that an unexpected bonus of additional high-grade leather can be produced. And there is less trimming when the stock is finished.

To check oxidation characteristics, rigid Fadeometer tests have been conducted over a period of time on various types of leathers fatliquored with the Polarsols. These bleached esterified oils proved to be unusually light fast. Also the Polarsols showed excellent stability where residual alum was present.

Perhaps one of the most attractive characteristics of these new esterified oils is the amount of money they save. According to Seaboard, the Polarsols soften leather to a greater degree than sulfated oils, so significant savings can be expected. Field experience has borne this out, with many tanners reporting they can get the same degree of softness in their stock with 20% less oil.

There is a Polarsol for every type of white leather—whether it be sides or splits, suedes or grains, kid or calf. The manufacturer offers percentage suggestions for use. To get detailed information and a trial sample of one of the Polarsols, specify your type of leather. Write or phone Seaboard Chemicals, Inc., Dept. 6G, 30 Foster St., Salem, Massachusetts, U. S. A. In Canada: Tennant & Michaud, Ltd., Toronto and Montreal.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